

An Investigation of the Aging Characteristics of FALANT Filters

George J. Kavarnos
Submarine Sonar Department



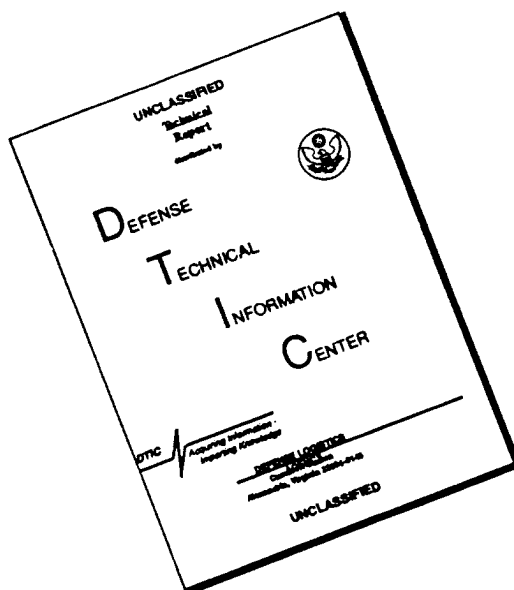
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PREFACE

The work described in this report was funded by the Naval Submarine Medical Research Laboratory, Naval Submarine Base New London, Groton, Connecticut (Project/Task 0933.002 W. U. 5404) under a work request entitled, "Evaluation of Color Filters for FALANT Filter Test," dated 18 July 1995.

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A handwritten signature in dark ink, appearing to read "R. J. Martin". The signature is fluid and cursive, with the first name "R." and last name "Martin" clearly distinguishable.

R. J. Martin
Head, Submarine Sonar Department

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AN INVESTIGATION OF THE AGING CHARACTERISTICS OF FALANT FILTERS

INTRODUCTION

The objective of this study was to determine the stability of plastic color filters used in the Farnsworth Lantern (FALANT) filter test. This project was performed for the Vision Department of the Naval Submarine Medical Research Laboratory in Groton, Connecticut, which uses the FALANT test for diagnosing color blindness.

The FALANT filters are polycarbonate or polyester plastics containing light refracting organic (aniline) dyes. The dyes are embedded or dispersed uniformly throughout the extruded plastic to ensure stability. The dyes are so chosen as to absorb a selected portion of the visible light spectrum. A primary red filter transmits radiant energy in the "red" portion of the spectrum but blocks other frequencies. The radiant energy that is absorbed is converted into heat. A green filter absorbs the "red" portion of the visible spectrum but allows passage of the "green" portion.

An important issue for the successful implementation of the FALANT filters concerns the stability of the filters in field testing. Since the plastic bases are exposed to light energy from a tungsten source, the dyes and plastics must be sufficiently stable to the absorbed heat, which, over a period of time, may degrade the filters. The absorbed heat can affect the plastic filters in two ways. First, heat can result in chemical changes in the dye and, therefore, in its transmission properties. Second, and perhaps of greater importance is the effect of heat on the migration or diffusion of the dye from the hot areas. Over a period of time, there will be a loss of dye from the filter resulting in the eventual fading of the color of the filter. Heat can also induce changes in the plastics themselves but, provided that they do not soften or melt, they can be expected to retain their stiffness. In general, acetate and vinyl plastics will soften at lower temperatures than will polyesters or polycarbonates, which are far more heat stable. Polyesters are heat stable to about 126° to 143° C; polycarbonates can be heated to 149° to 163° C without noticeable softening. However, polycarbonate filters are more difficult and expensive to produce and require a special technology to combine with the dye.

Another potential source of deterioration in the filters may be photochemistry resulting from exposure to the light. Since many of the dyes used in the filters absorb visible light, the possibility of photochemical degradation must also be taken into account. Photochemistry is more likely to occur at the "blue" end of the visible spectrum and below 400 nm, in the near ultraviolet. In this portion of the spectrum, dyes containing unsaturated bonds can absorb radiant light energy and, if exposed to light radiation over a period of time, may experience photochemical degradation.

Given this background, a test plan was devised to evaluate the stability of some selected filters (table 1) under various conditions. Initial spectroscopic studies employing Fourier transform infrared spectroscopy (FTIR) and ultraviolet/visible (UV/VIS) spectrophotometry were carried out to determine the spectral characteristics of several classes of filters, including vinyl, polyester, and polycarbonate formulations. These filters were exposed to heat, humidity, and light over extended periods of time. Upon completion of the incubation times, the spectroscopic studies were repeated, and the "final" spectra compared with the original. Any changes in the plastics or the dyes after exposure to heat, humidity, or light would be detected and monitored by changes in the FTIR and UV/VIS spectra.

EXPERIMENTAL

BASELINE SPECTROSCOPIC STUDIES

Infrared Spectroscopy

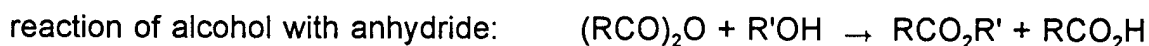
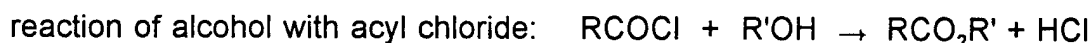
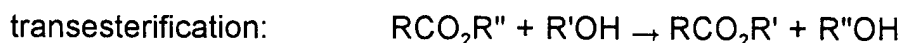
FTIR was used to determine the infrared characteristics of each filter formulation. This methodology was chosen because FTIR is much faster and much more versatile in chemical structure elucidation than conventional infrared spectroscopy. An infrared spectrometer produces infrared light from a radiation source that is focussed and passed through the sample. The transmitted light is then dispersed by a monochromator into component wavelengths that are eventually converted into a signal. The FTIR measurements were performed on a Nicolet 510 FTIR spectrometer. The system used in this study used a computer package called OMNIC to process the data.

The infrared region consists of wavelengths between 4000 cm^{-1} (2.5 microns) and 650 cm^{-1} (15.4 microns). In this region of the electromagnetic spectrum, almost any chemical compound possessing covalent bonds will absorb radiation.¹ In two different molecules, covalent bonds have different natural frequencies of vibration, depending on the nature of the atoms making the bond and the structure of the molecules, and, consequently, their patterns of absorption in the infrared region, where the frequencies may match bond stretching and bending, will not be identical. Infrared spectroscopy can thus be used to great advantage to identify and characterize the structure of molecules.

Each FALANT filter was inserted into the path of the laser light of the infrared spectrometer and the infrared spectrum of each filter was recorded. The spectra obtained typically pertained to the structures of the polymers used to embed the dyes. The concentrations of the dyes in the plastic filters were too low and below the limit of detection. For purposes of structure elucidation, the infrared spectrum of each plastic

filter was compared with a spectrum from a polymer library contained in the OMNIC software. The infrared spectra together with the spectra resulting from the library are shown in figures 1 to 12. The salient structural characteristics of the polymers used in each filter and the rationale in selecting the "best" match from the polymer library are discussed below.

Rosco Cinegel plastic filters 3402, 3403, 3404, and 3415 will be considered here as a group since their spectral properties are quite similar. These plastic samples were labeled as "polyesters." Polyesters are a versatile and diverse group of polymers used as plastic, fibers, and coatings.² They are synthesized by reaction between an organic acid, acyl chloride, or anhydride with an alcohol:



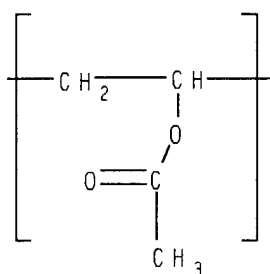
The salient structural feature of a polyester is the ester group, which consists of a carbonyl group (carbon bonded to an oxygen atom) adjacent to an oxygen. The ester group links together organic units such as aromatic rings or methylene chains to form the polymer. The methylene units account for the "plastic" properties of polyesters. On the other hand, the inclusion of aromatic rings can produce "chain stiffening." The relative amounts of the aromatic and methylene groups thus determine the unique thermal and mechanical properties of this class of polymers.

Rosco Cinegel plastic filters - 3402, 3403, 3404, and 3415 - display infrared spectra that match closely with the spectra of poly(vinyl acetate), vinyl alcohol/vinyl acetate copolymer, and several polyesters from the polymer library included in the OMNIC software package. An analysis of the spectral patterns can help to identify the composition of these samples. Starting with the high wavenumbers (cm^{-1}) on the left of the spectrum, we can draw several conclusions. First, the region near 3500 cm^{-1} is where oxygen-hydrogen bonds (OH), which are characteristic of alcohols, display absorption. Since the absorption in this region in the plastic samples is too weak, we can rule out polymers containing OH functionality such as the vinyl alcohol/vinyl acetate copolymer. Normally, the absorption in this region is broad, suggesting the presence of extensive hydrogen bonding in this polymer. Second, the broad peak at wavenumbers to the right of 3000 cm^{-1} is indicative of absorptions due to stretches on carbon-hydrogen bonds in saturated or completely aliphatic molecular systems. The presence of weak absorption slightly to the left of 3000 cm^{-1} is important because this

implies the presence of stretching vibrations due to carbon-hydrogen bonds connected to aromatic (ring) systems.

Another observation of importance is the peak at about 1730 cm^{-1} which appears in each of the spectra discussed in this section. This peak, due to the carbonyl absorption, is diagnostic of the ester group. Further evidence for the ester group is suggested by the strong absorptions between 1300 and 1000 cm^{-1} , which are due to carbon-oxygen stretches.

Samples 3402, 3403, 3404, and 3415 appear to match most closely with the spectra of vinyl alcohol/vinyl acetate copolymers, poly(vinyl acetate) and polyesters such as poly(1,4-butylene terephthalate). Vinyl alcohol/vinyl acetate copolymers can be ruled out because, as mentioned earlier, these polymers usually exhibit broad absorption due to an oxygen-hydrogen stretch, which does not appear in the spectra of the filters. A second possibility, poly(vinyl acetate), is, strictly speaking, not a polyester, but instead has a repeating vinyl group in the polymer backbone with acetate side chains:



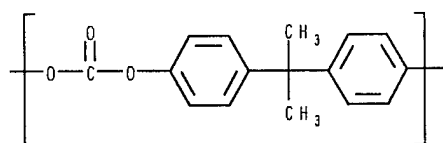
The spectra of samples 3402, 3403, 3404, and 3415 appear to match more closely the spectrum of poly(1,4-butylene terephthalate) because of the presence of the peak at about 750 cm^{-1} , which is not present in the spectrum of poly(vinyl acetate). This peak may be due to out-of-plane bending vibrations characteristic of aromatic compounds. We conclude that these plastic filters are most likely fabricated from a polyester prepared by condensation of a linear glycol and an organic acid such as terephthalic acid.

Similar considerations apply to the Roscolux plastics labeled as "tough, white, diffusion" polyesters. These include samples 111, 116, 117, and 118. The spectra of these plastics are all strongly suggestive of a linear polyester similar to samples 3402, 3403, 3404, and 3415.

Kodak 2556 plastic film represents a special case because its spectrum reveals broad absorption between 3000 and 3500 cm^{-1} suggesting the presence of hydrogen-bonding. Although the remaining portion of the spectrum appears more complex than

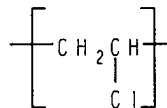
the simpler polyesters discussed earlier, it nonetheless has retained certain features indicative of the polyesters. The presence of the 750 cm^{-1} peak again rules out that this plastic is a non-aromatic material such as vinyl alcohol/vinyl acetate copolymer. It is more likely a polyester containing, perhaps, the terephthalic group blended with poly(vinyl alcohol).

Plastics 122 and 339 were designated as polycarbonates and indeed their spectra match very nicely the spectrum of a polycarbonate resin from the OMNIC spectral library. Although, strictly speaking, a polycarbonate belongs to the polyester class, it possesses unique material properties that differ in several respects from conventional linear polyesters. The polymer backbone consists of repeating units of carbonic acid and a dihydroxy compound such as a bisphenol:²



The salient structural features of the polycarbonate plastic can be deduced from the infrared spectra. The absorption to the left and right of 3000 cm^{-1} clearly indicates the presence of aliphatic and aromatic carbon-hydrogen stretches. This is consistent with the presence of aromaticity in the polymer. The most likely candidate is the bisphenol unit, which is used in numerous polycarbonate formulations. The absorptions between 700 and 900 cm^{-1} also confirm aromaticity. The peak between 1700 and 1800 cm^{-1} is due to carbonyl absorption.

Finally, sample 871 gives an infrared spectrum that matches that of a poly(vinyl chloride). This polymer, which is composed of repeating units of vinyl chloride, differs from the polyesters and polycarbonates discussed above:



In figure 12, since there is no absorption to the left of 3000 cm^{-1} , the presence of

aromaticity can be ruled out. The absorption bands between 600 and 800 cm^{-1} are probably due to carbon-chlorine bond stretches. There is little absorption between 1000 and 1300 cm^{-1} , which would be due to carbon-oxygen stretches in ester-like materials. The peak at about 1700 cm^{-1} is too weak to designate as carbonyl absorption, although the plastic may contain small amounts of an additive containing a carbonyl group.

Ultraviolet/Visible (UV/VIS) Spectroscopy

UV/VIS spectroscopy is very similar in principle and methodology to infrared spectroscopy, with the exception that shorter wavelengths are used to scan the material. Ultraviolet and visible spectra typically have less detail than infrared, so that less structural information can be gleaned from a UV/VIS study. The visible portion of the spectrum includes wavelengths from about 400 to 700 nm. The ultraviolet region extends from about 180 to 400 nm. The spectra of each plastic, prior to incubation, were obtained using the portion of the spectrum from 300 to 700 nm. The wavelengths between 300 and 400 nm are collectively dubbed the "near" ultraviolet region. The spectra were recorded using a Perkin-Elmer Model Lambda 14 UV/VIS Spectrometer. The following settings were used: scan speed - 240.00 nm/min; data interval - 1.00 nm; and slit width - 2.00 nm.

The UV/VIS spectra of the plastics analyzed in this study are shown in figures 13 to 24. As these figures show, all of the plastics are transparent to visible light. However, there are subtle differences in the spectra that can be ascribed to the different dyes dispersed in the filters, as the polymers themselves are colorless and should not absorb visible light radiation. The colored plastics display absorption patterns in the visible region. For example, sample 339 shows strong absorption below 600 nm. This accounts for the pinkish color of this plastic. Sample 122, labeled as Roscolux green diffusion polycarbonate, is transparent at about 525 nm. On either side of 525 nm, there is strong absorption.

Accordingly, the UV/VIS spectroscopic study was expected to give information concerning the stability of the dyes dispersed in each plastic. If indeed there were environmental effects influencing the dyes in the plastic, these effects would be indicated by changes in the UV/VIS spectra of each plastic.

INCUBATION CONDITIONS

The plastics were cut into three separate pieces before exposure to the following environmental conditions: (1) incubation at 38°C for 34 days; (2) incubation

at 75% relative humidity for 34 days; and (3) continuous exposure to visible light radiation for several days.

A temperature of 38°C was deliberately chosen to avoid excessive heating of the plastics. Previous testing performed by the Stereo Optical Co. indicated that the lantern's 40-watt bulb did not heat the filters excessively. The plastics were at all times during exposure maintained well below their melting and softening temperatures. The polyesters are reported to be heat stable up to temperatures ranging from 126° to 143°C.³ The polycarbonates are even more heat stable - they do not deform until heated to 149° to 163°C. Accordingly, the objective of performing the aging study at 38°C was to determine the stability of both the polymers (using IR spectroscopy) and the dyes (using UV/VIS spectroscopy) over time. This test was carried out in a Despatch Series 900 oven.

The effect of humidity on the plastics was carried out in a large desicator jar containing a saturated sodium chloride (NaCl) solution. The relative humidity in the jar is about 75%.⁴

The objective of the light exposure study was to determine if light radiating from the tungsten source degrades the filters during their operational lifetime. The 40-watt bulb used in the FALANT lantern was placed in the center of a 190 x 100 mm Pyrex glass dish. The plastic filters were positioned along the perimeter of the dish on the inside wall. This configuration corresponds to a distance of 85 mm (about 3.75 in.) from the lamp to the filters. Although it was impossible to determine the exact number of hours of light exposure because the lamp burnt out unexpectedly over a weekend, we can estimate a lower bound of 3 days and an upper bound of 7 days. During incubation, there was no noticeable build-up of heat in the plastic filters, confirming earlier observations made at the Naval Submarine Medical Research Laboratory.

FINAL SPECTROSCOPIC STUDIES

Following the incubation times specified in the previous section, the spectroscopic studies were repeated to detect aging effects. The IR spectra are shown in figures 25 to 35 (due to experimental difficulties, the IR spectrum for sample 118 was not recorded). For comparison, these figures also show the original or baseline spectra. In all cases, the spectra are practically identical. The slight differences noted in some cases are probably within experimental error and need not be considered here. If changes in the polymer were taking place during incubation, we would expect differences in the IR spectra. Since the spectra are, for the most part, identical, the polymers are most likely unaffected by the temperature, humidity, and light exposure during incubation.

Similarly, the UV/VIS spectra recorded after the incubation times were identical.

These spectra are shown in figures 36 to 47, where they have been superimposed on the baseline spectra. Again, there are no observable differences. This observation strongly suggests that the dyes are stable and do not undergo chemical changes or migration from the plastics during incubation.

CONCLUSIONS

There are at least two mechanisms to be considered as possible causes of aging in the plastic filters used in the Falant test lantern: (1) chemical modification to the dyes or plastics and (2) migration or loss of the dye during incubation. This study, however, has shown quite conclusively that heating the plastics at 38°C for a prolonged period of time does not lead to any observable change in polymer composition or in the concentration of the dye. This is not a totally unexpected conclusion, however. The temperature of the plastics was maintained well below their melting points. Close to the melting points, the plastics begin to soften and change dimensionally. However, the incubation temperature, 38°C, was deliberately chosen to be sufficiently below the melting point to avoid any significant degradation due to heating. This was confirmed by the similarities of the IR spectra obtained before and after incubation for all samples.

Do the dyes migrate and diffuse during the 38°C incubation? Probably not. The rates of migration will ultimately influence the rate of fading of the color filters. The UV/VIS study shows that aging due to migration and diffusion does not occur at 38°C during the time of incubation used in this study. Consequently, the dye is probably well dispersed and stable within the plastic filters, since otherwise the UV/VIS spectra would show enhanced transmission in the visible region, which was not observed.

Humidity was shown by this study to have a negligible effect on the aging characteristics of the filters. The IR and UV/VIS spectra of all filters before and after incubation at 75% relative humidity were practically identical. The poly(ethylene terephthalate) polyesters in this study are known to be water insoluble, which is consistent with their stability under humid conditions.⁵

Exposure to light from the 40-watt bulb used in the Falant lantern can, in principle, lead to changes due to light absorption (photochemistry) or to heating effects. Photochemistry can be due to light absorption by either the polymer matrix or the embedded dye. If photochemistry is taking place, it is likely to be due to light absorption by the dyes rather than the polymers. The plastic polymers used in the Falant test are refractive to light in the visible range, so any *a priori* major photochemical process involving the polymers can be excluded. The dyes, however, do absorb in the visible. But the near identity of the UV/VIS spectra (which measure the absorption

peaks due to the dyes) before and after exposure to light confirm the absence of any photochemistry involving the dyes.

Heating effects due to light absorption can also be ruled out. The amount of heat absorbed by the filter depends on the identity of the dye, i. e., "green" filters absorb more of the "red" visible spectrum and, accordingly, may suffer more heat damage than "red" filters, which transmit red light. But the IR spectra of sample 122, labeled as a "green diffusion polycarbonate" (before and after heat incubation) are nearly identical, again underscoring the stability to light of these filters.

In conclusion, the plastic filters manufactured by Rosco Labs display long-term stability to heat, humidity, and visible light. Although the conditions employed in this study were not "drastic," they do represent realistically the conditions of long-term usage and storage of the plastic filters. The incubation times selected for this study were somewhat arbitrary. Still, they were sufficiently long to be able to conclude that the temperature, humidity, and light conditions used in this study were not sufficient to degrade the transmission properties of the plastics.

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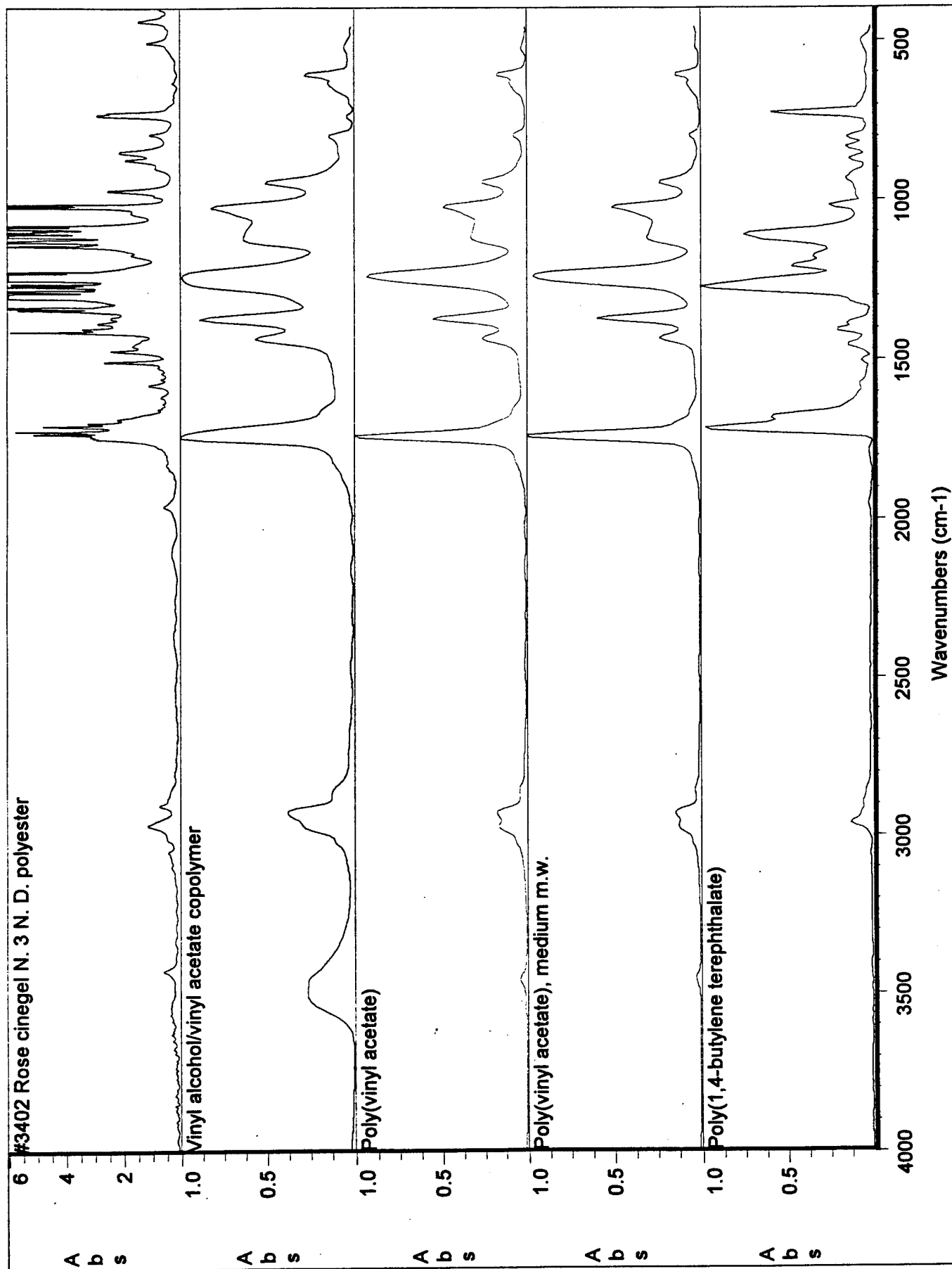


Figure 1. Infrared Spectra of Filter 3402 and of Matches from Library Search.

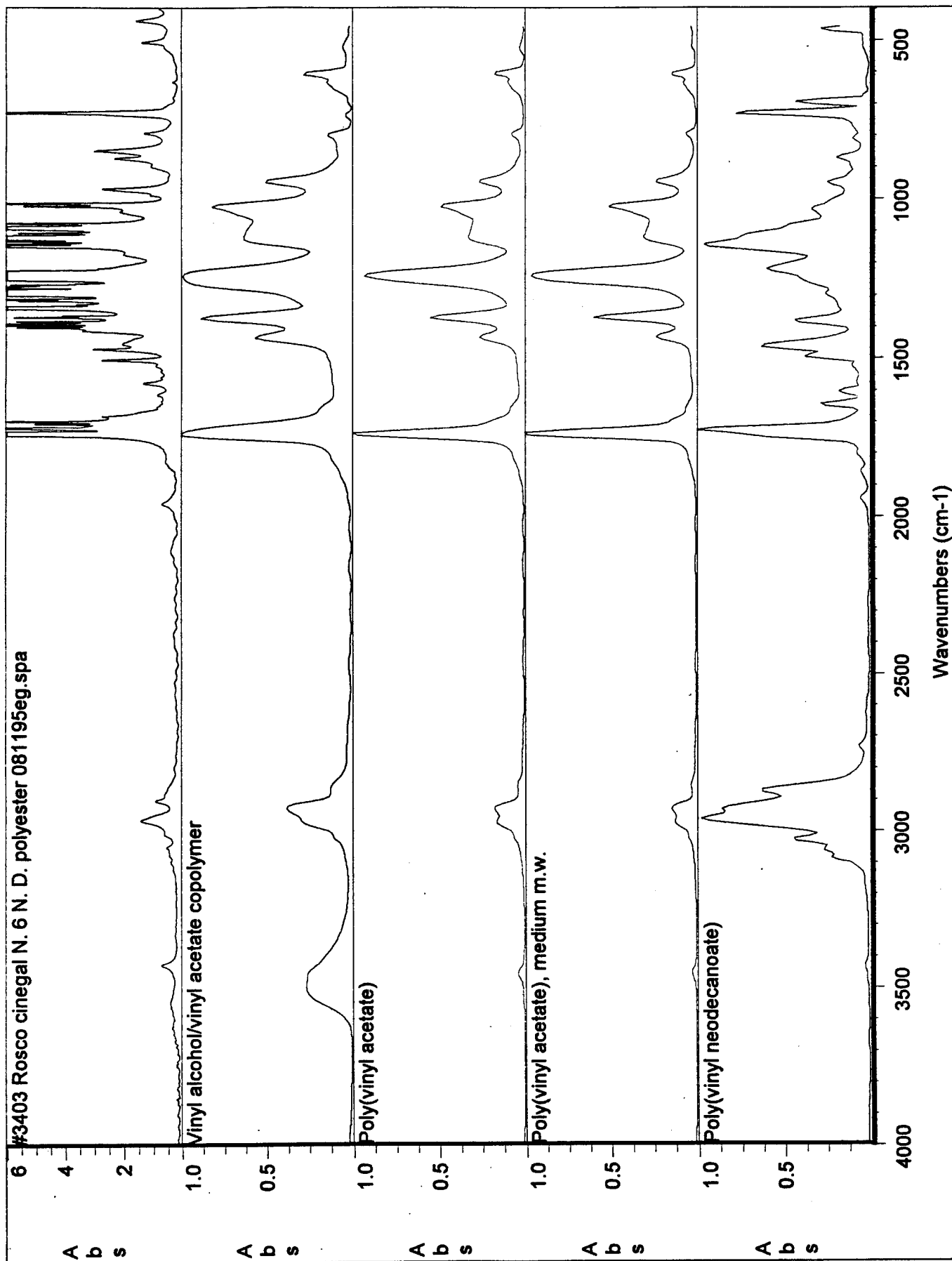


Figure 2. Infrared Spectra of Filter 3403 and of Matches from Library Search.

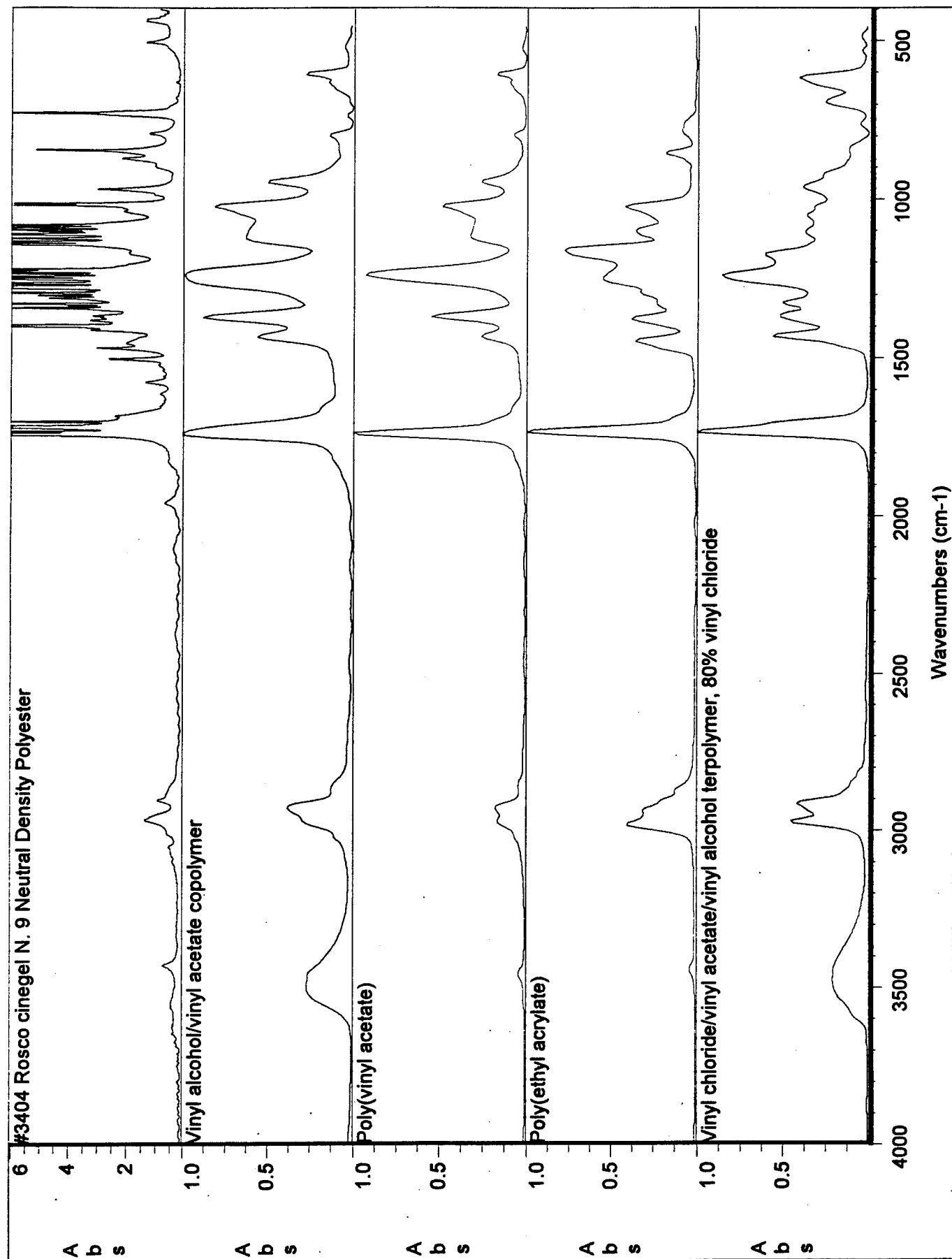


Figure 3. Infrared Spectra of Filter 3404 and of Matches from Library Search.

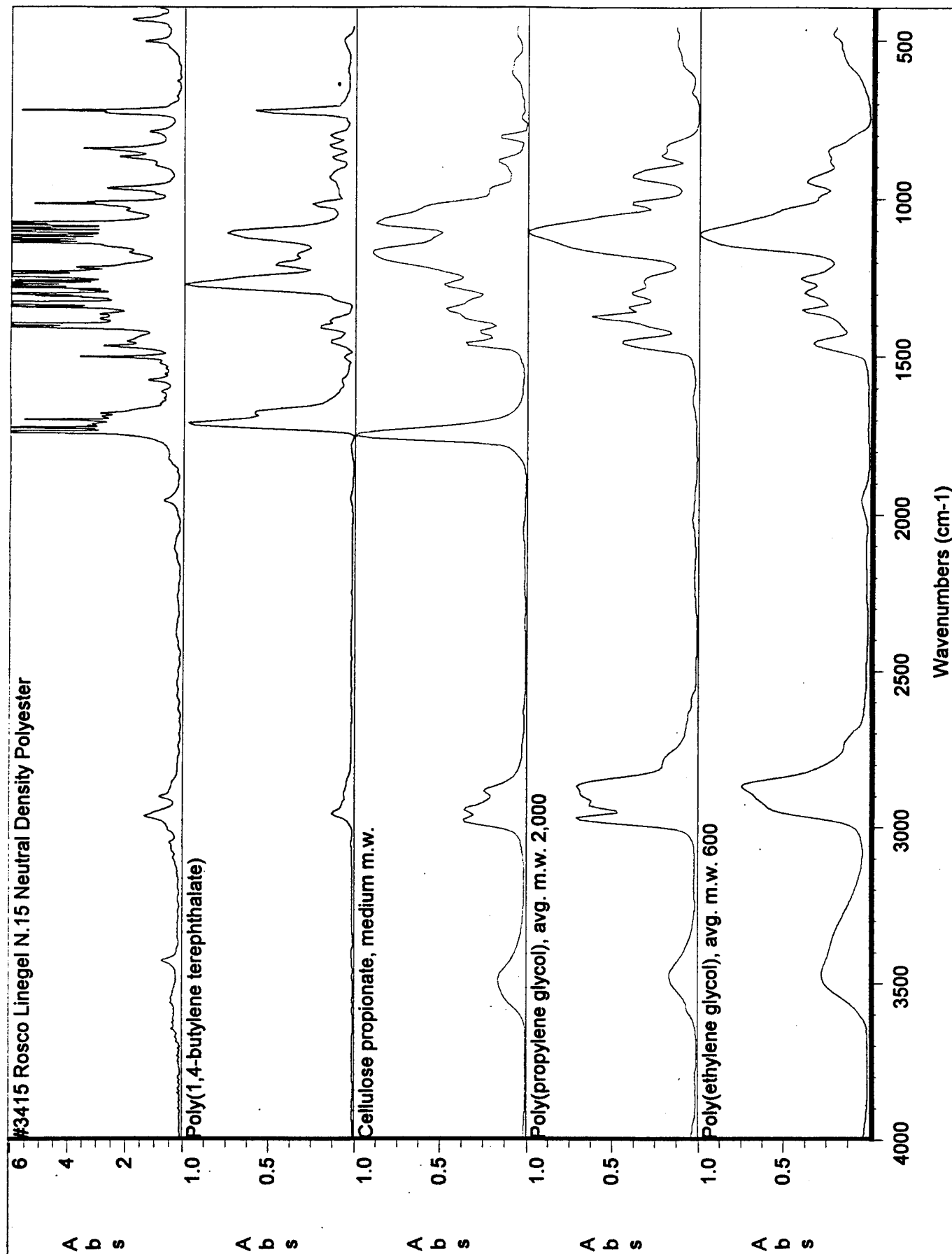


Figure 4. Infrared Spectra of Filter 3415 and of Matches from Library Search.

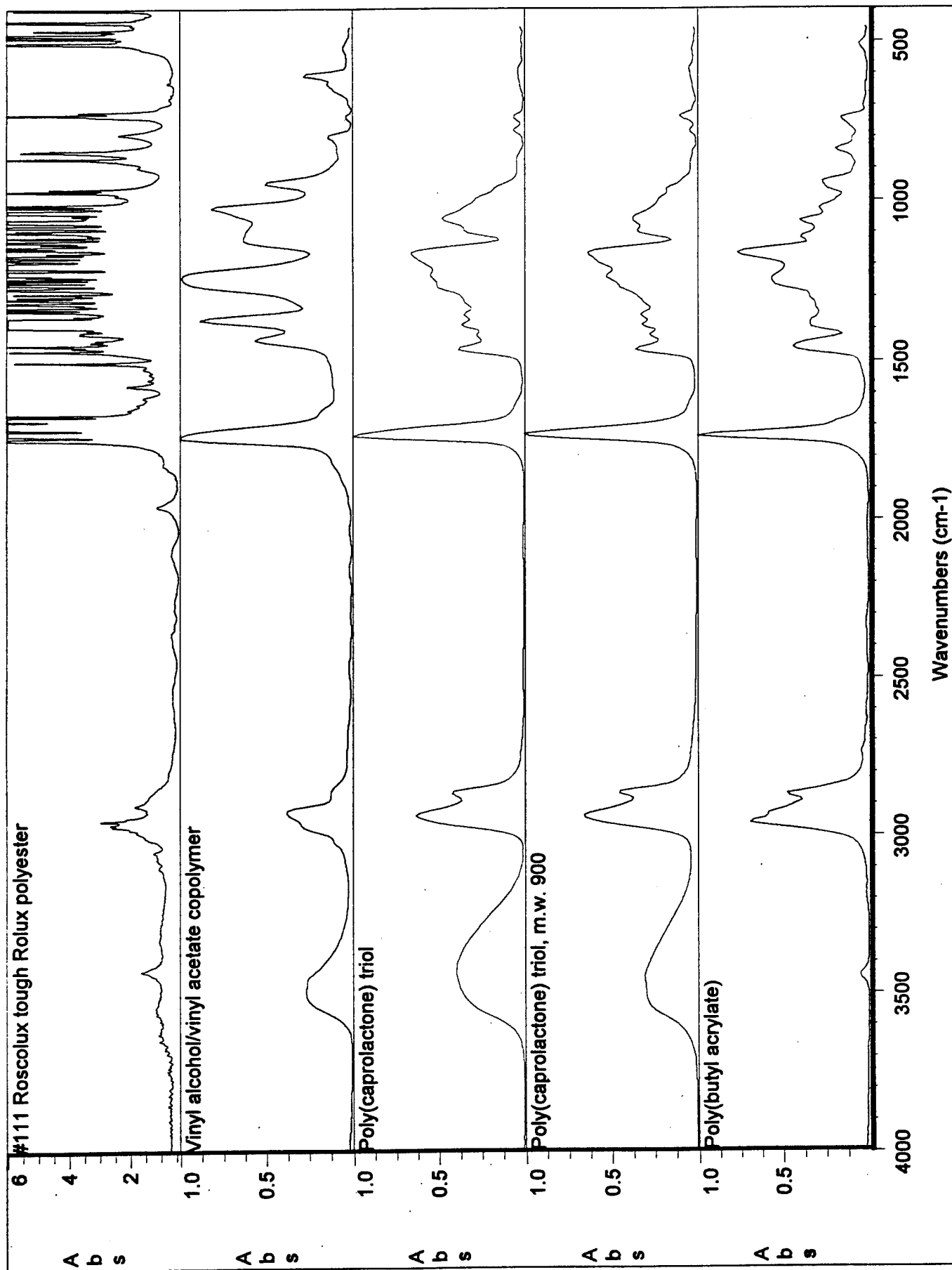


Figure 5. Infrared Spectra of Filter 111 and of Matches from Library Search.

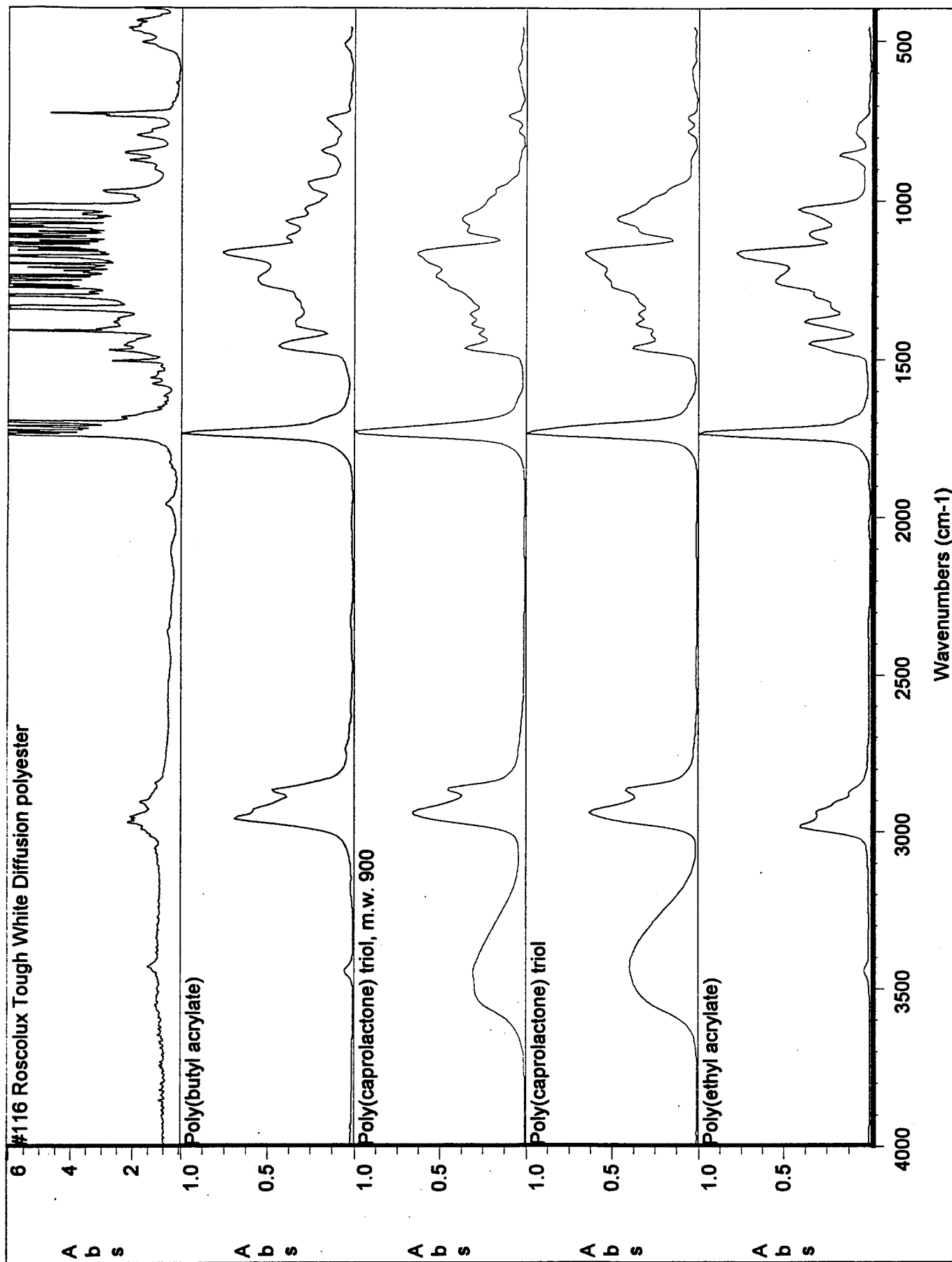


Figure 6. Infrared Spectra of Filter 116 and of Matches from Library Search.

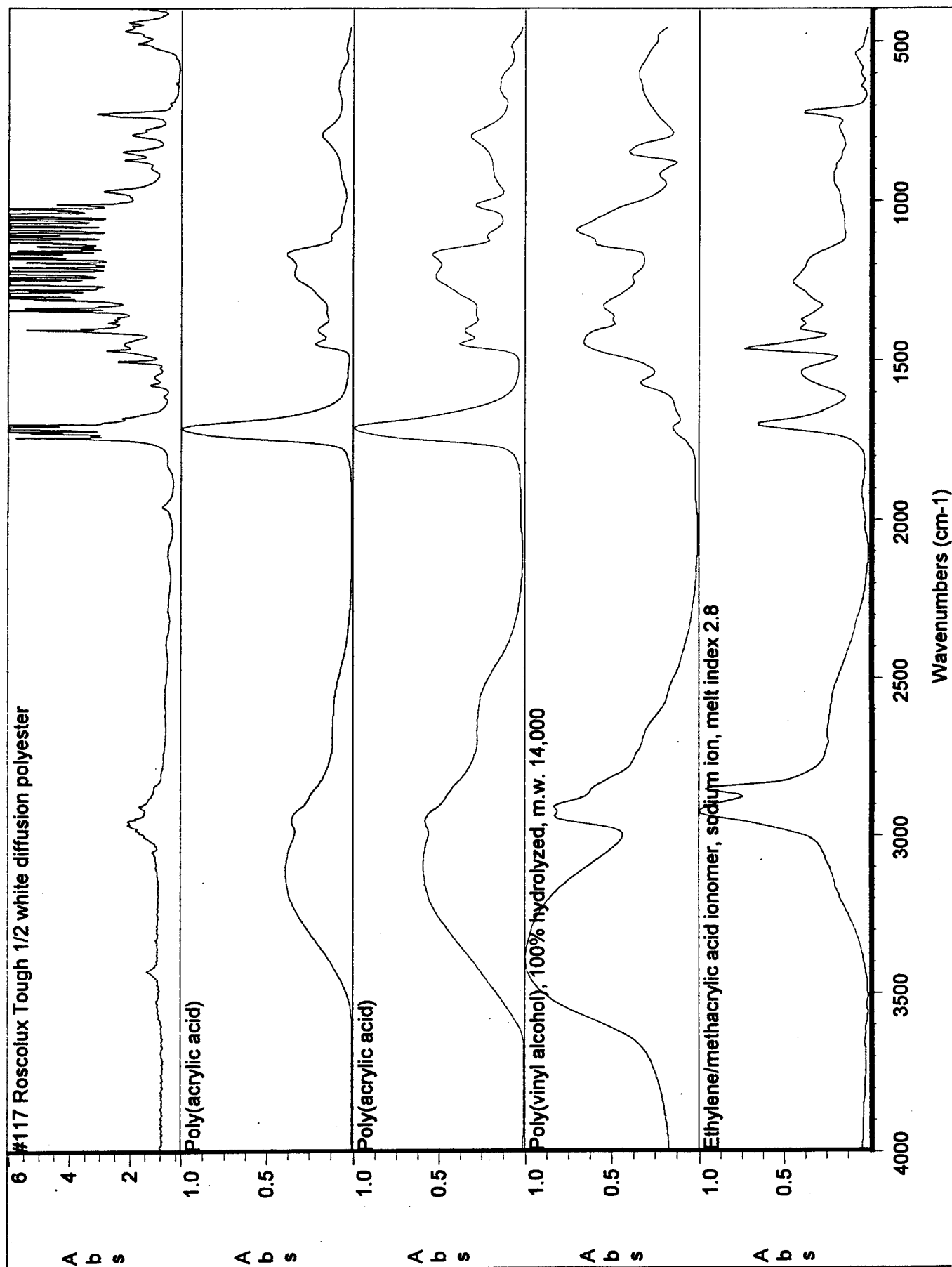


Figure 7. Infrared Spectra of Filter 117 and of Matches from Library Search.

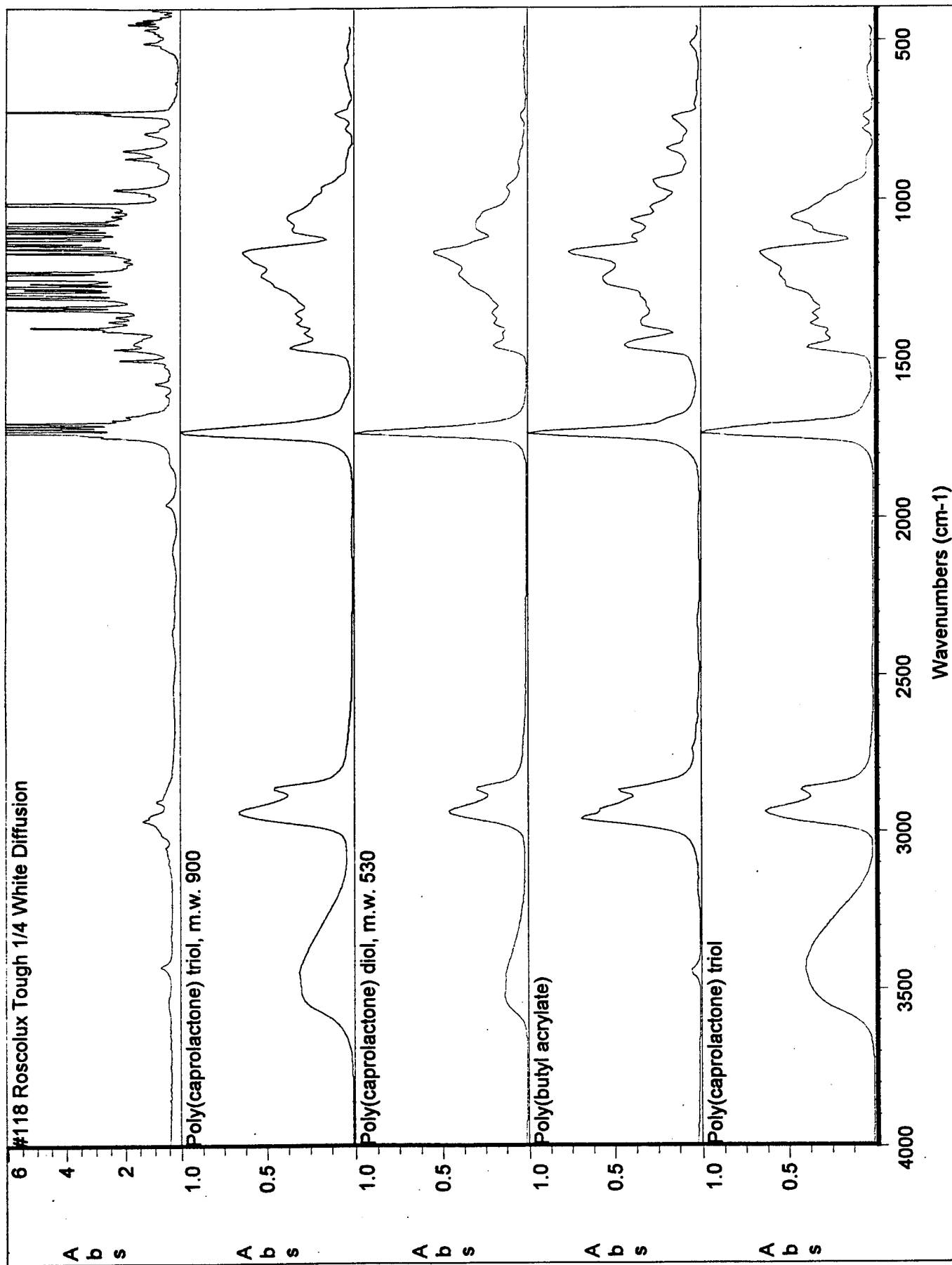


Figure 8. Infrared Spectra of Filter 118 and of Matches from Library Search.

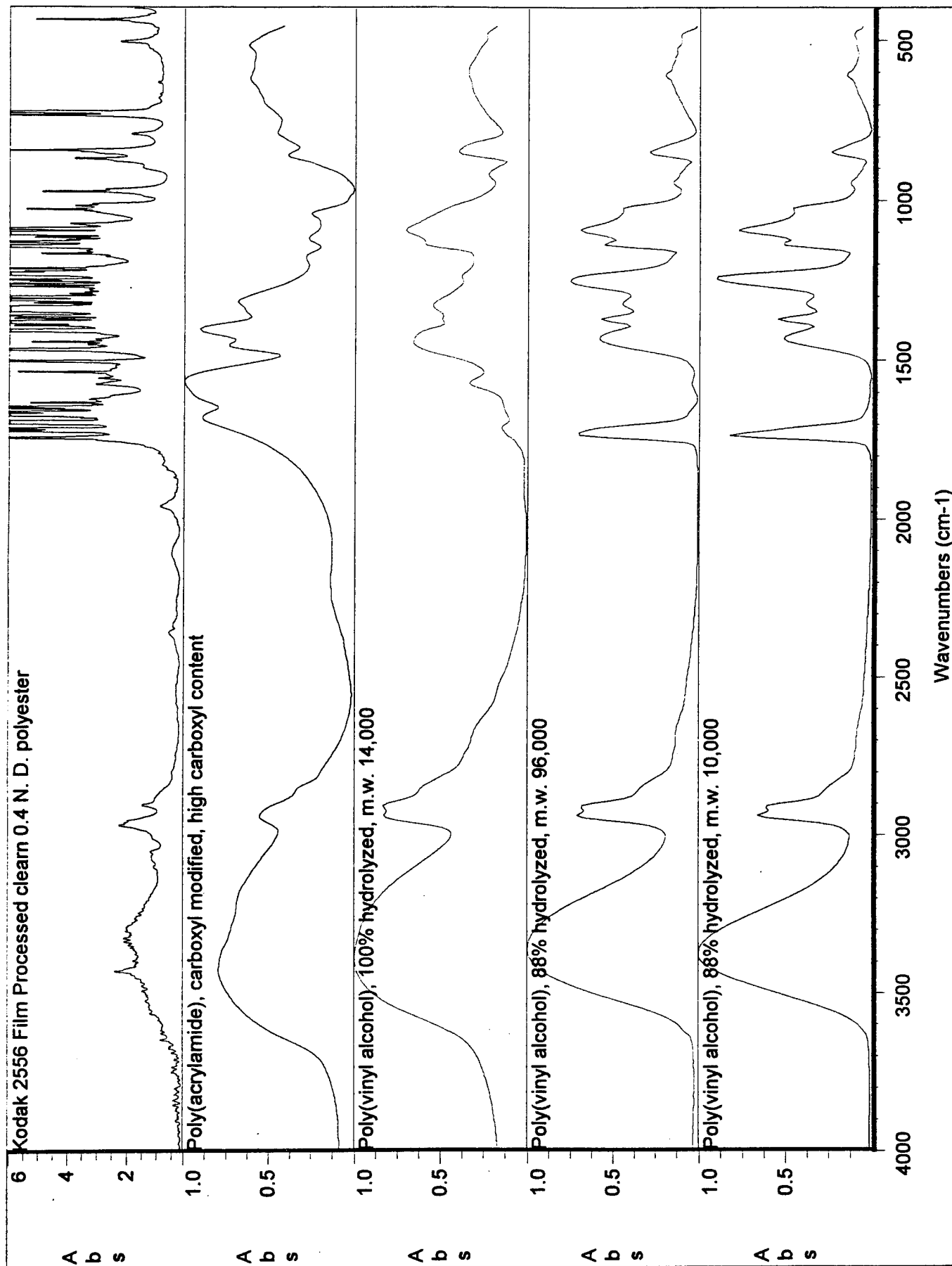


Figure 9. Infrared Spectra of Filter 2556 and of Matches from Library Search.

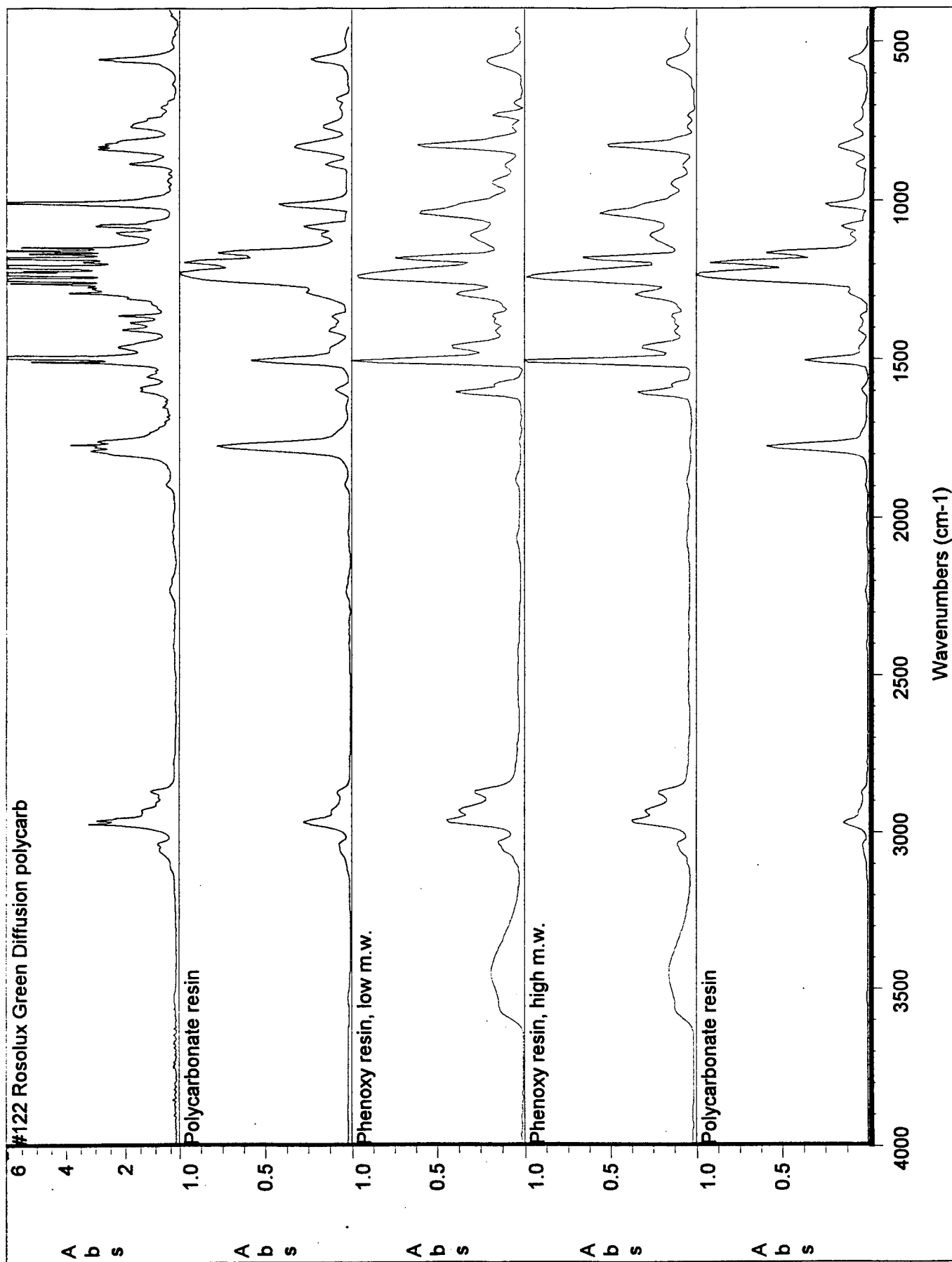


Figure 10. Infrared Spectra of Filter 112 and of Matches from Library Search.

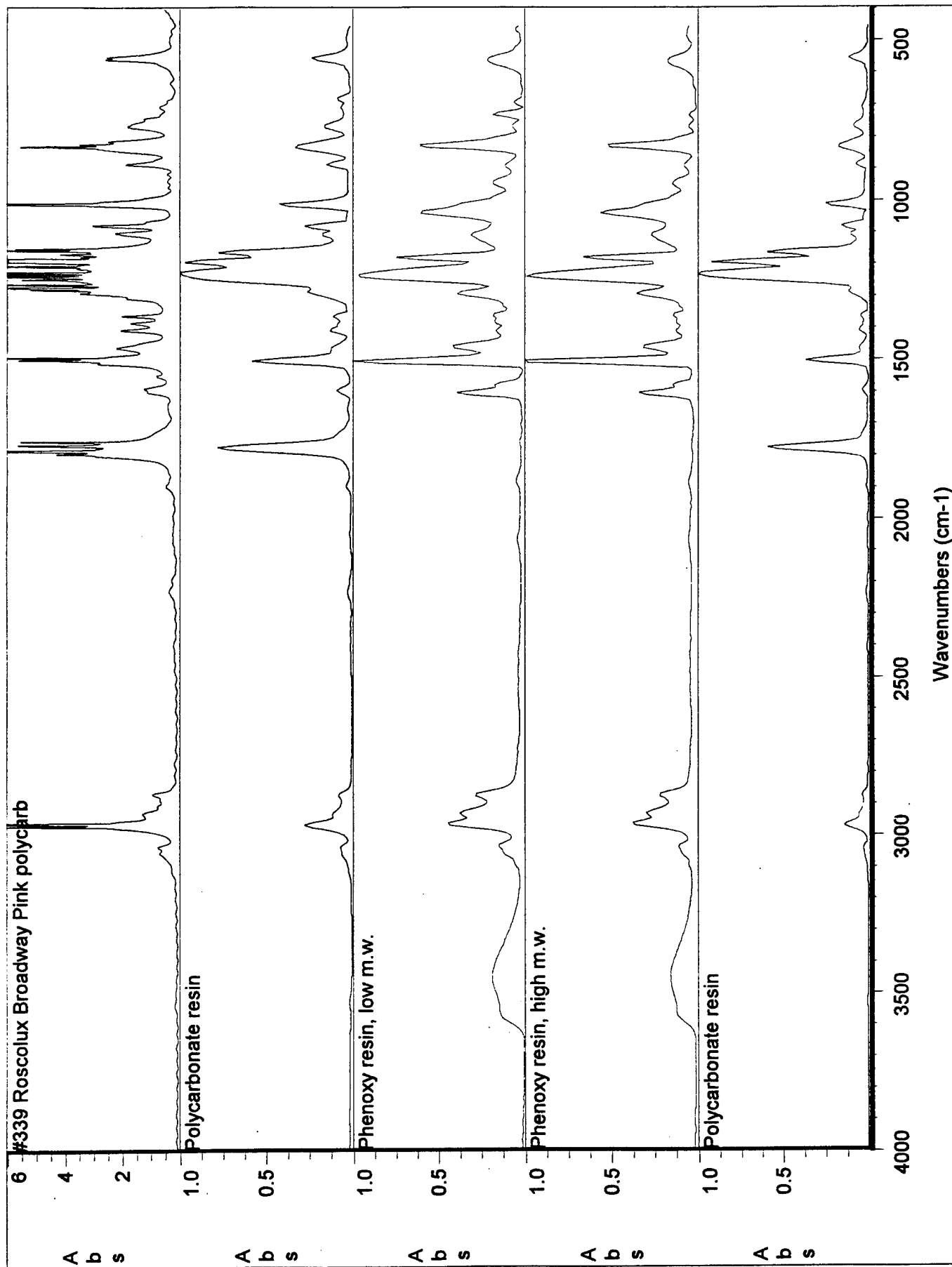


Figure 11. Infrared Spectra of Filter 339 and of Matches from Library Search.

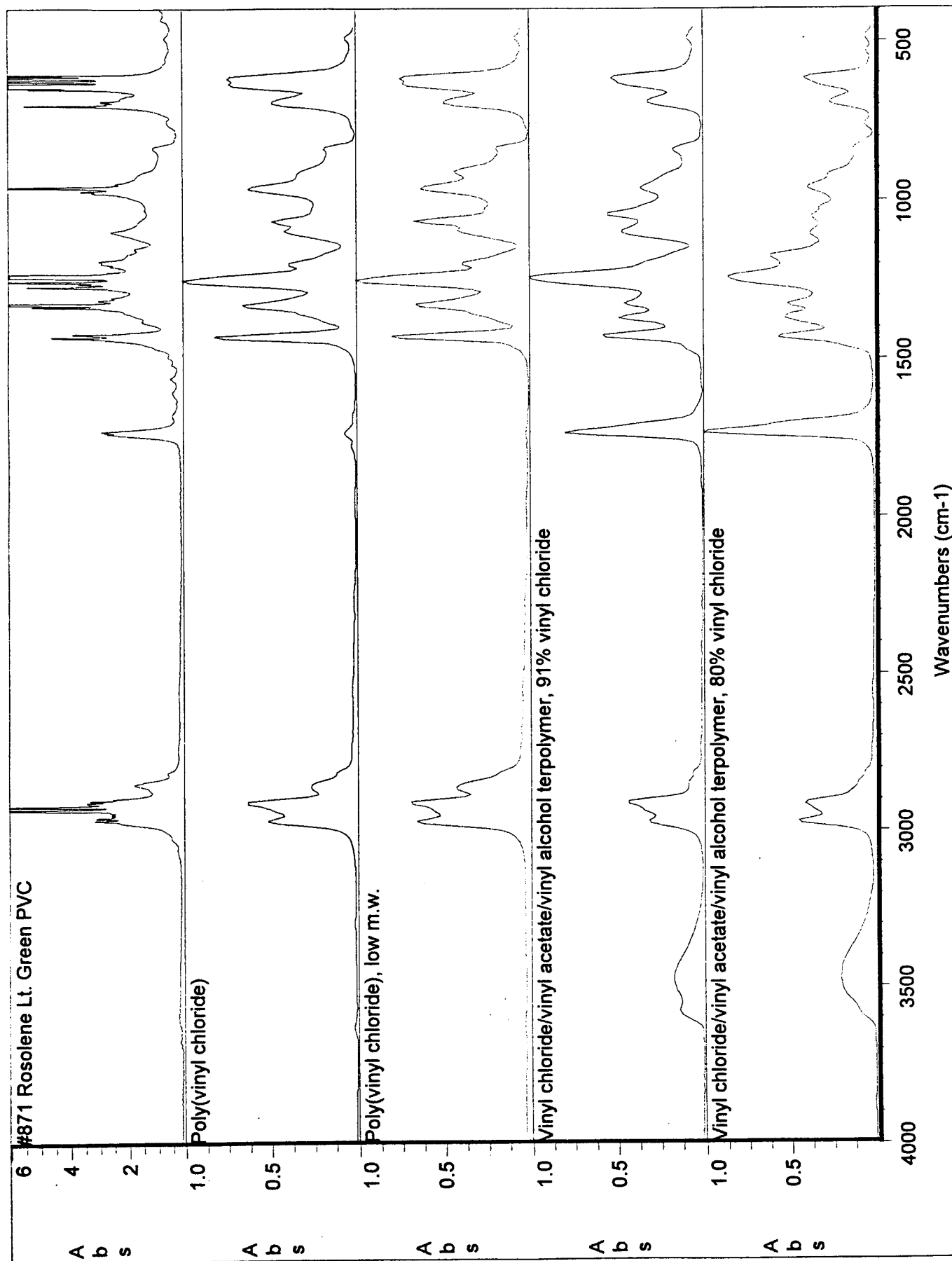


Figure 12. Infrared Spectra of Filter 871 and of Matches from Library Search.

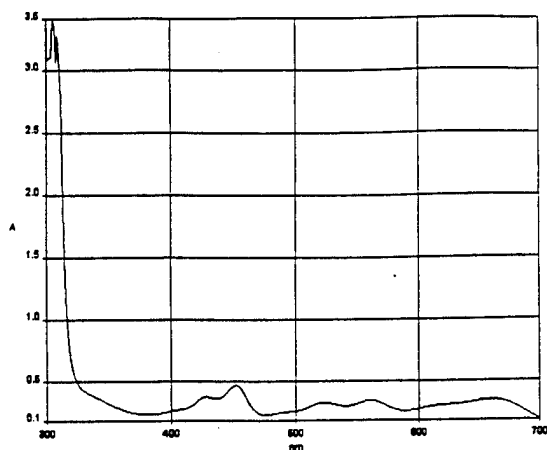


Figure 13. UV/VIS Spectrum of Filter 3402 Before Incubations.

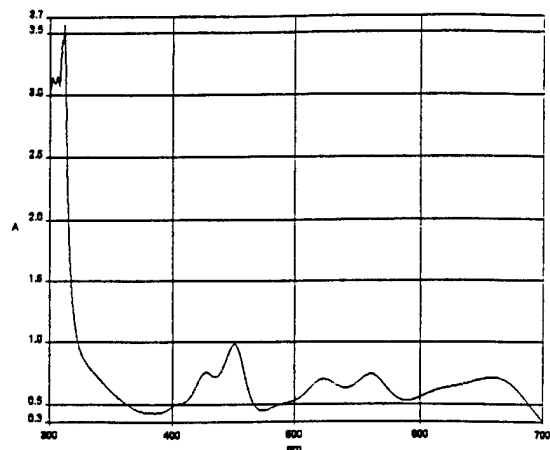


Figure 14. UV/VIS Spectrum of Filter 3403 Before Incubations.

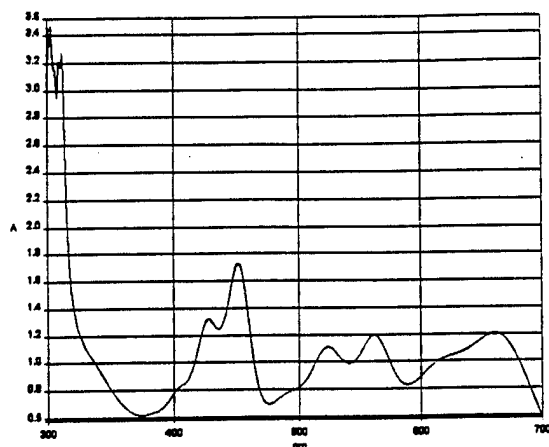


Figure 15. UV/VIS Spectrum of Filter 3404 Before Incubations.

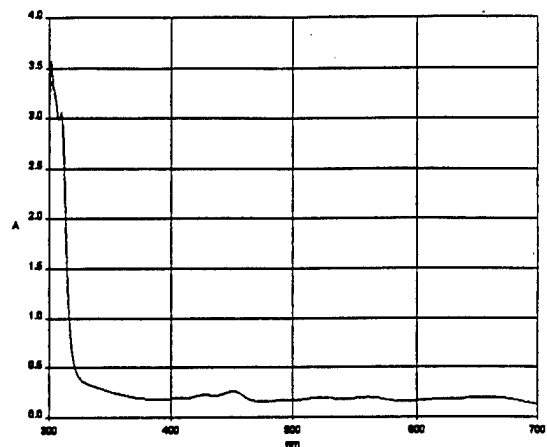


Figure 16. UV/VIS Spectrum of Filter 3415 Before Incubations.

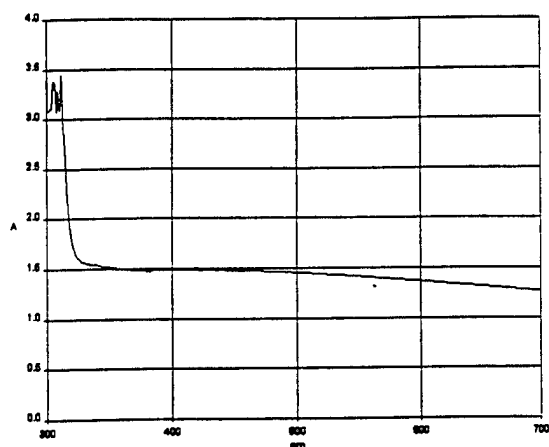


Figure 17. UV/VIS Spectrum of Filter 111 Before Incubations.

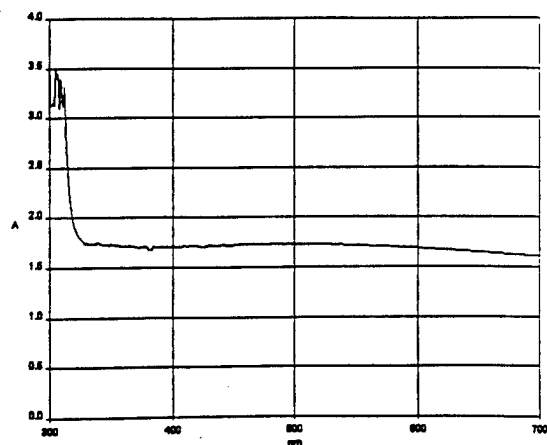


Figure 18. UV/VIS Spectrum of Filter 116 Before Incubations.

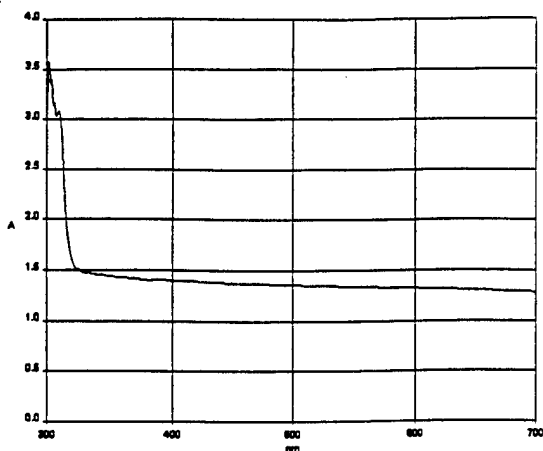


Figure 19. UV/VIS Spectrum of Filter 117 Before Incubations.

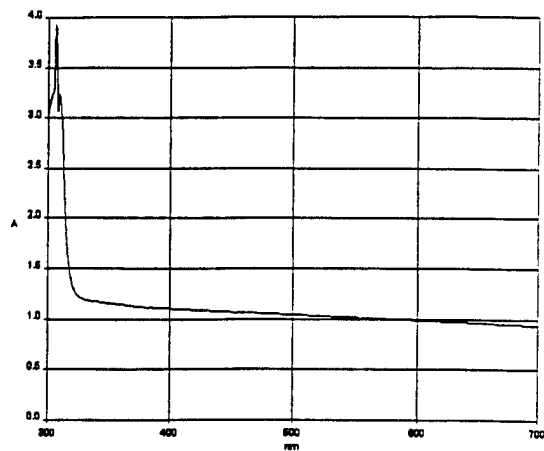


Figure 20. UV/VIS Spectrum of Filter 118 Before Incubations.

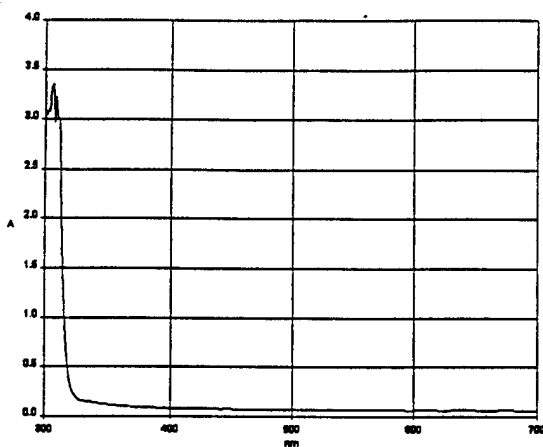


Figure 21. UV/VIS Spectrum of Filter 2556 Before Incubations.

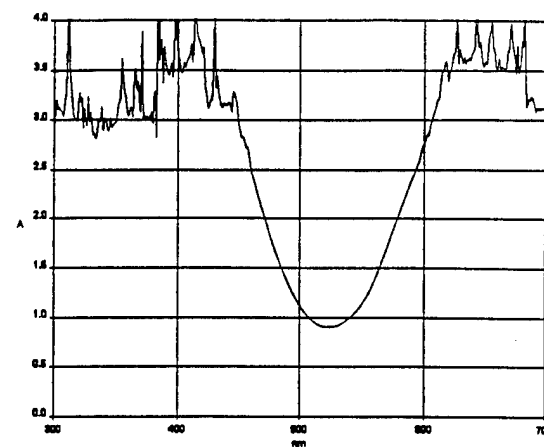


Figure 22. UV/VIS Spectrum of Filter 122 Before Incubations.

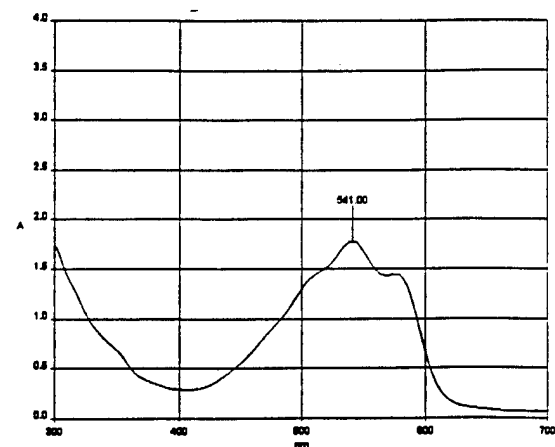


Figure 23. UV/VIS Spectrum of Filter 339 Before Incubations.

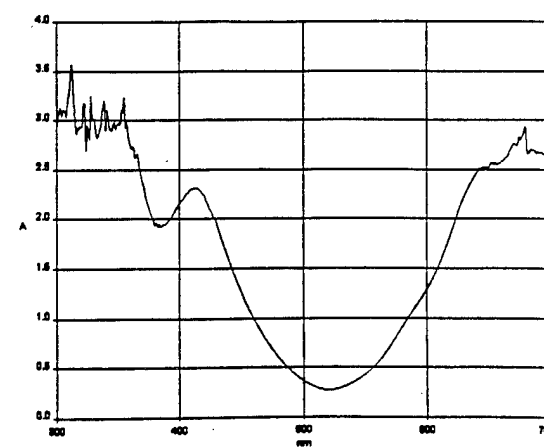


Figure 24. UV/VIS Spectrum of Filter 871 Before Incubations.

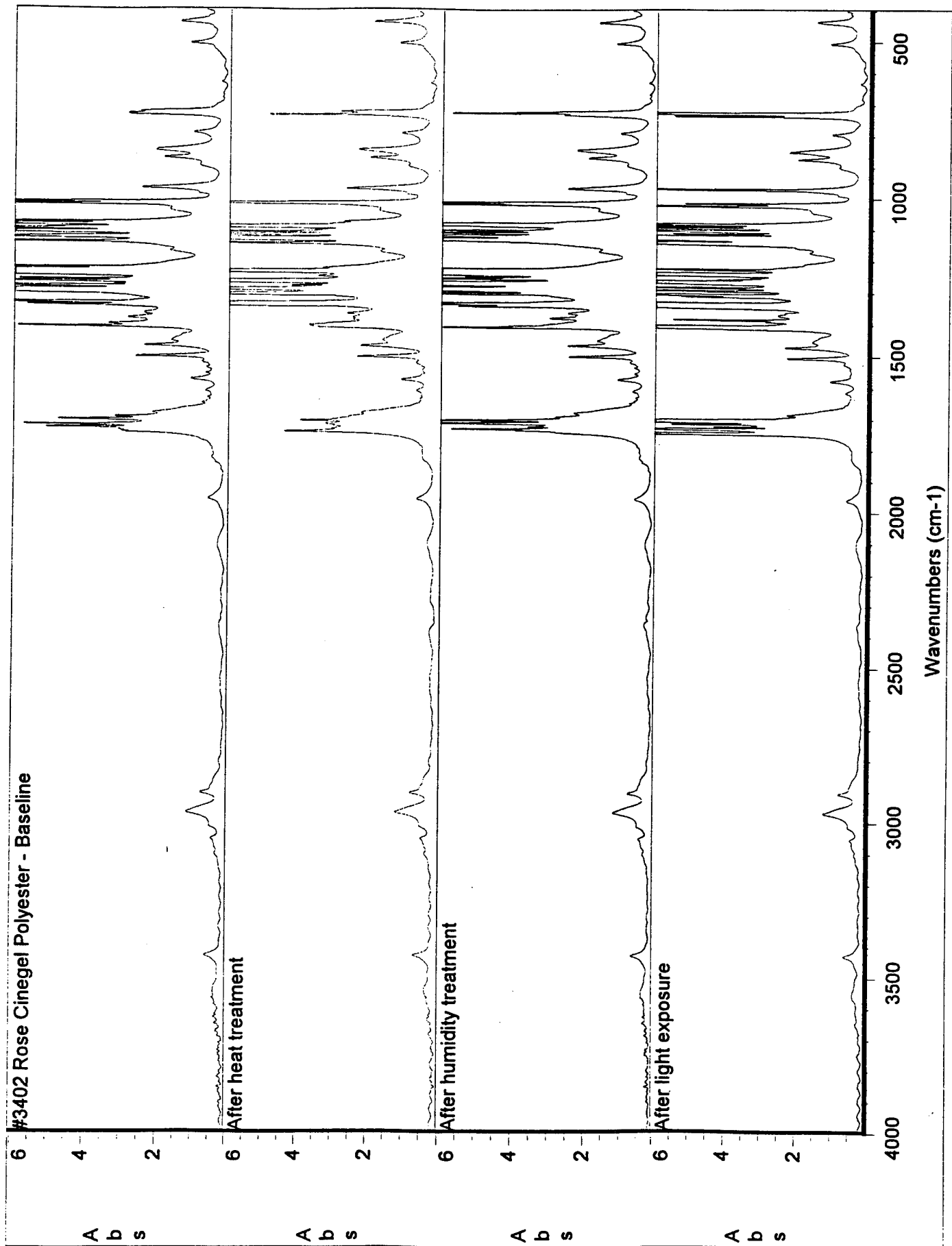


Figure 25. Infrared Spectrum of Filter 3402 After Incubations.

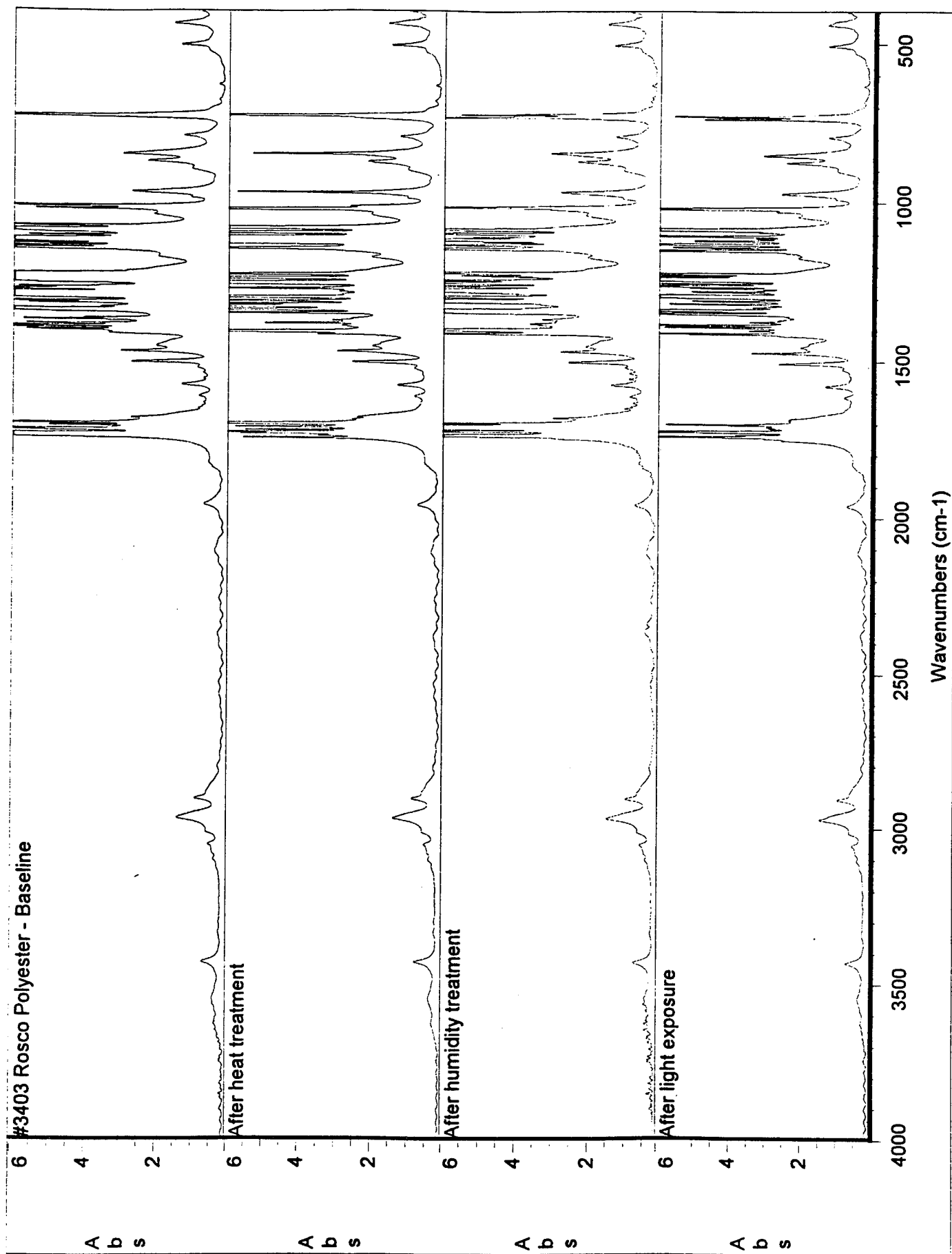


Figure 26. Infrared Spectrum of Filter 3403 After Incubations.

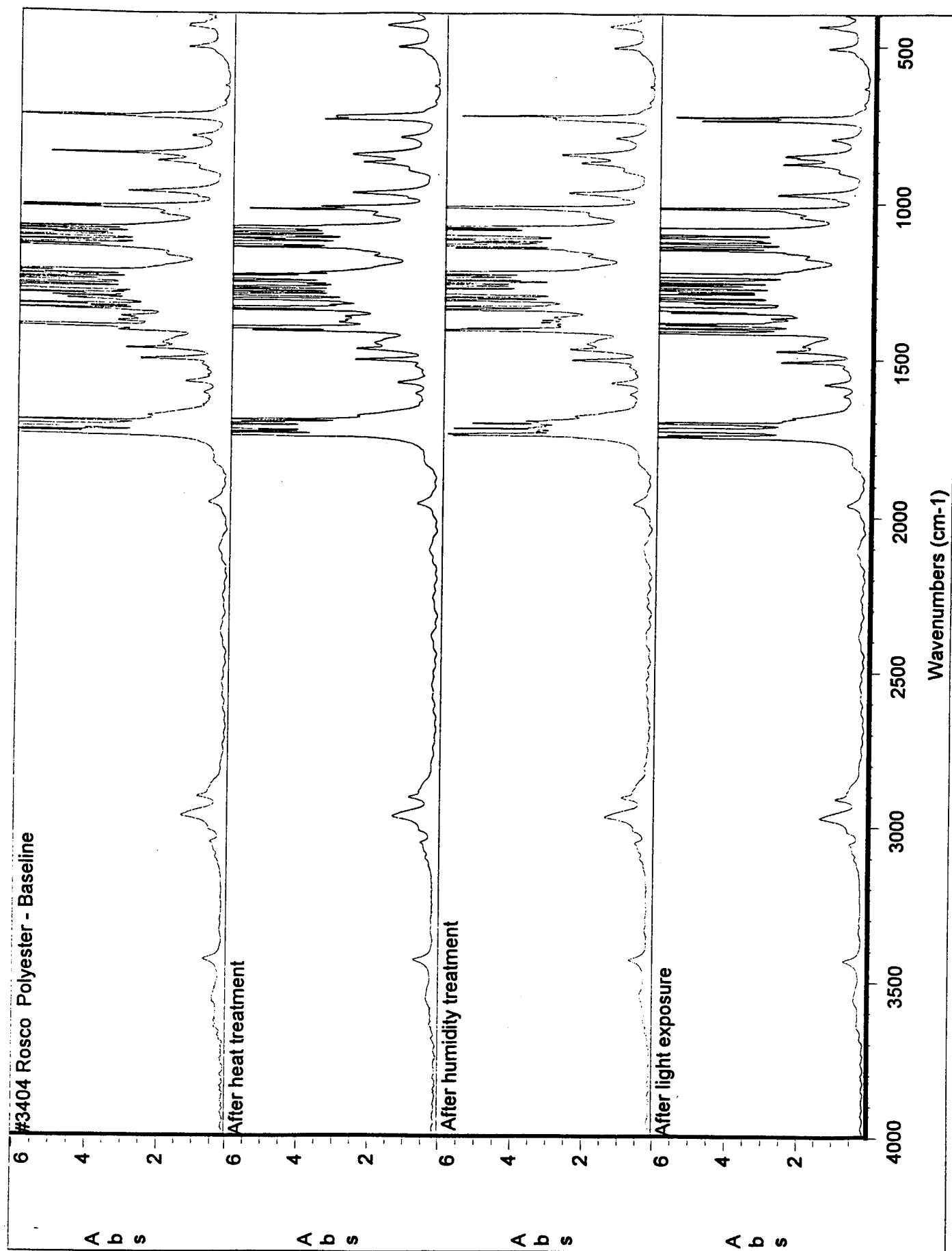


Figure 27. Infrared Spectrum of Filter 3404 After Incubations.

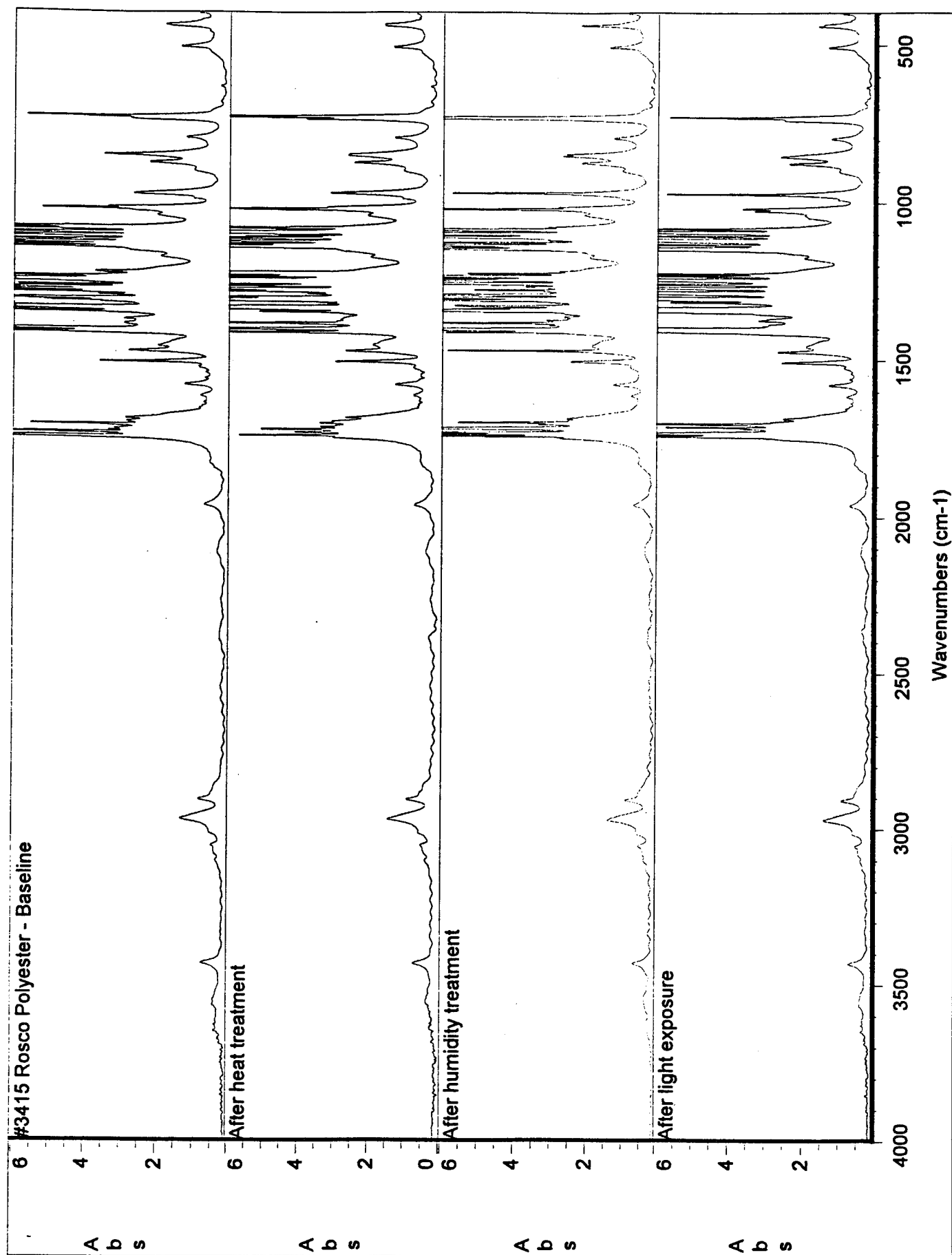


Figure 28. Infrared Spectrum of Filter 3415 After Incubations.

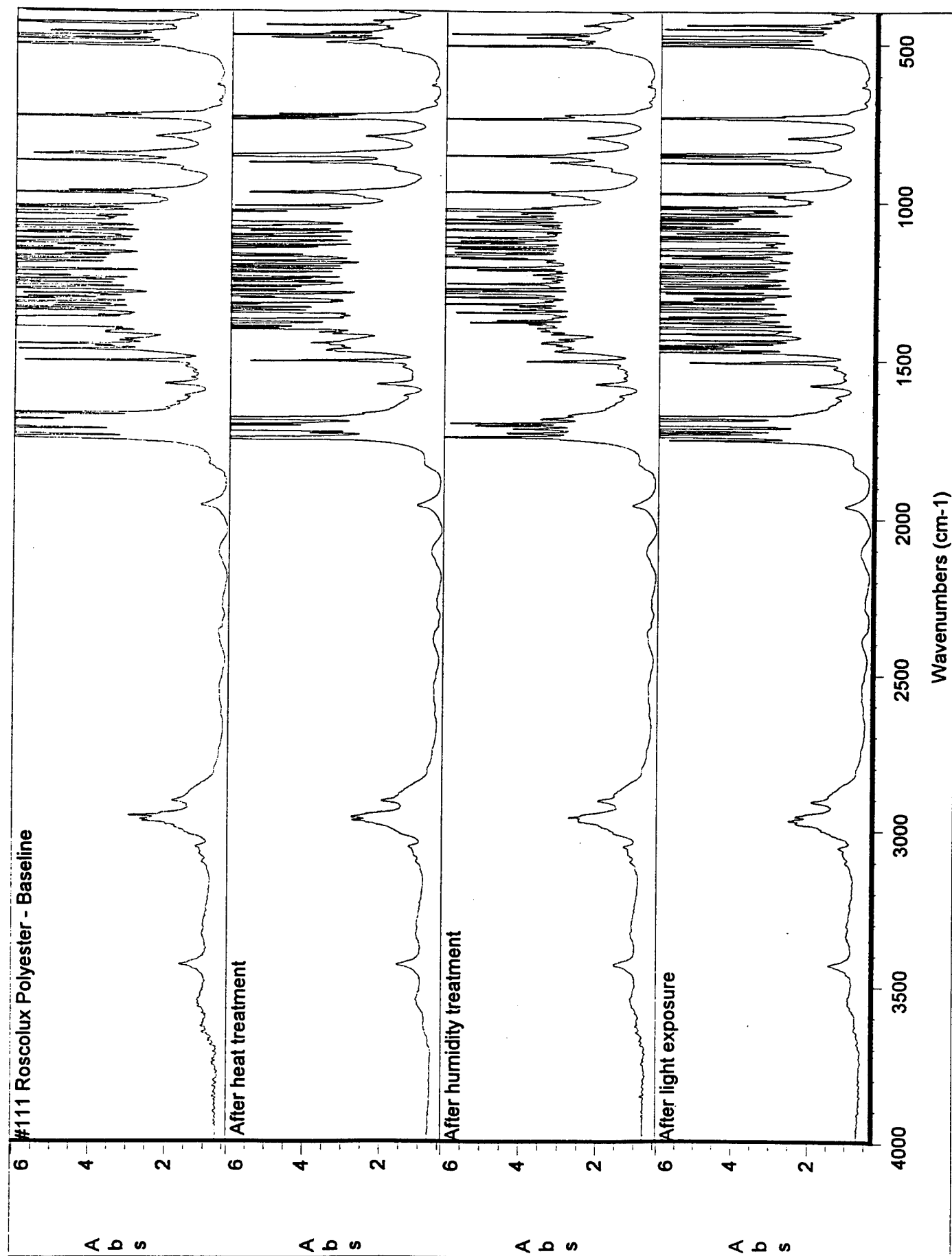


Figure 29. Infrared Spectrum of Filter 111 After Incubations.

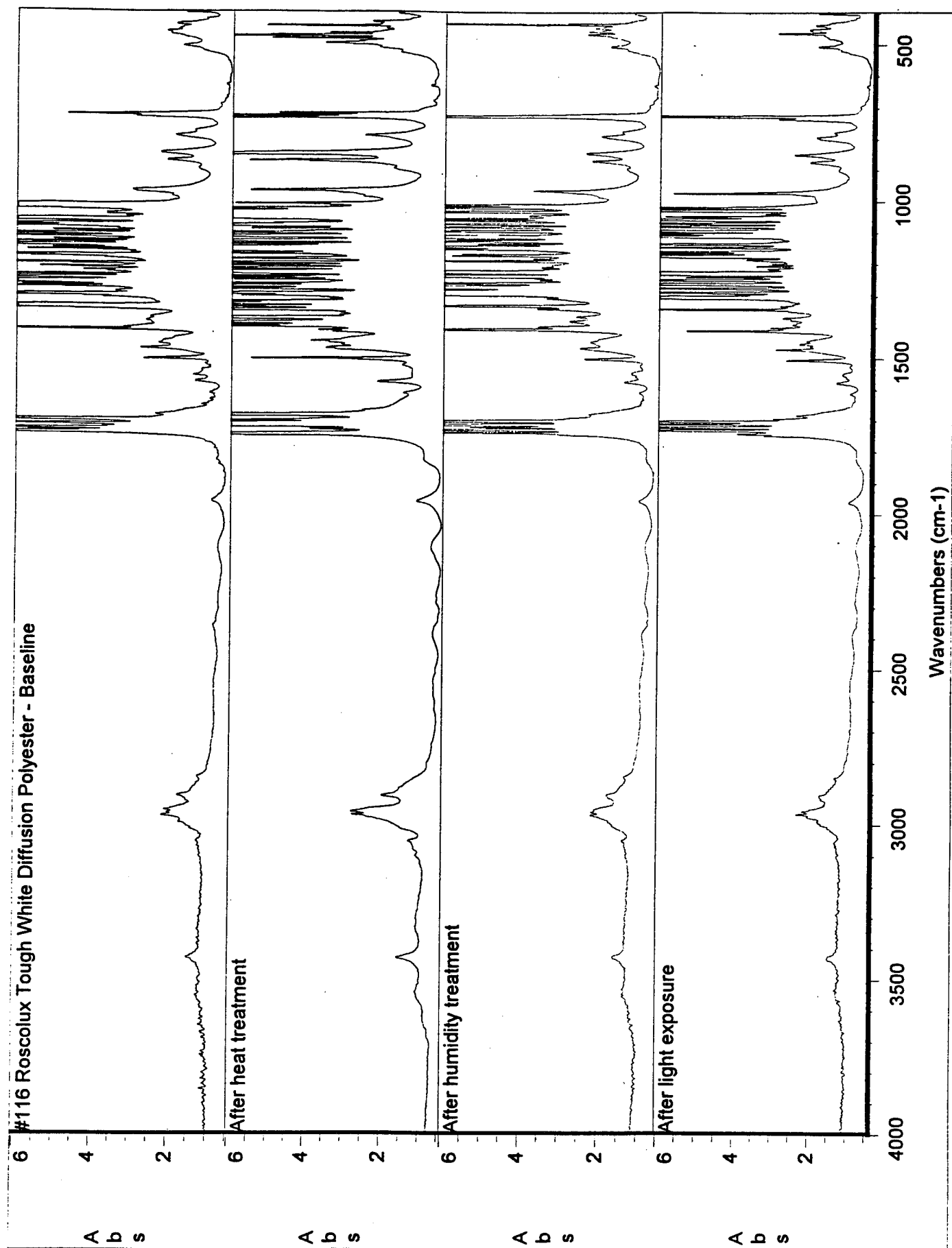


Figure 30. Infrared Spectrum of Filter 116 After Incubations.

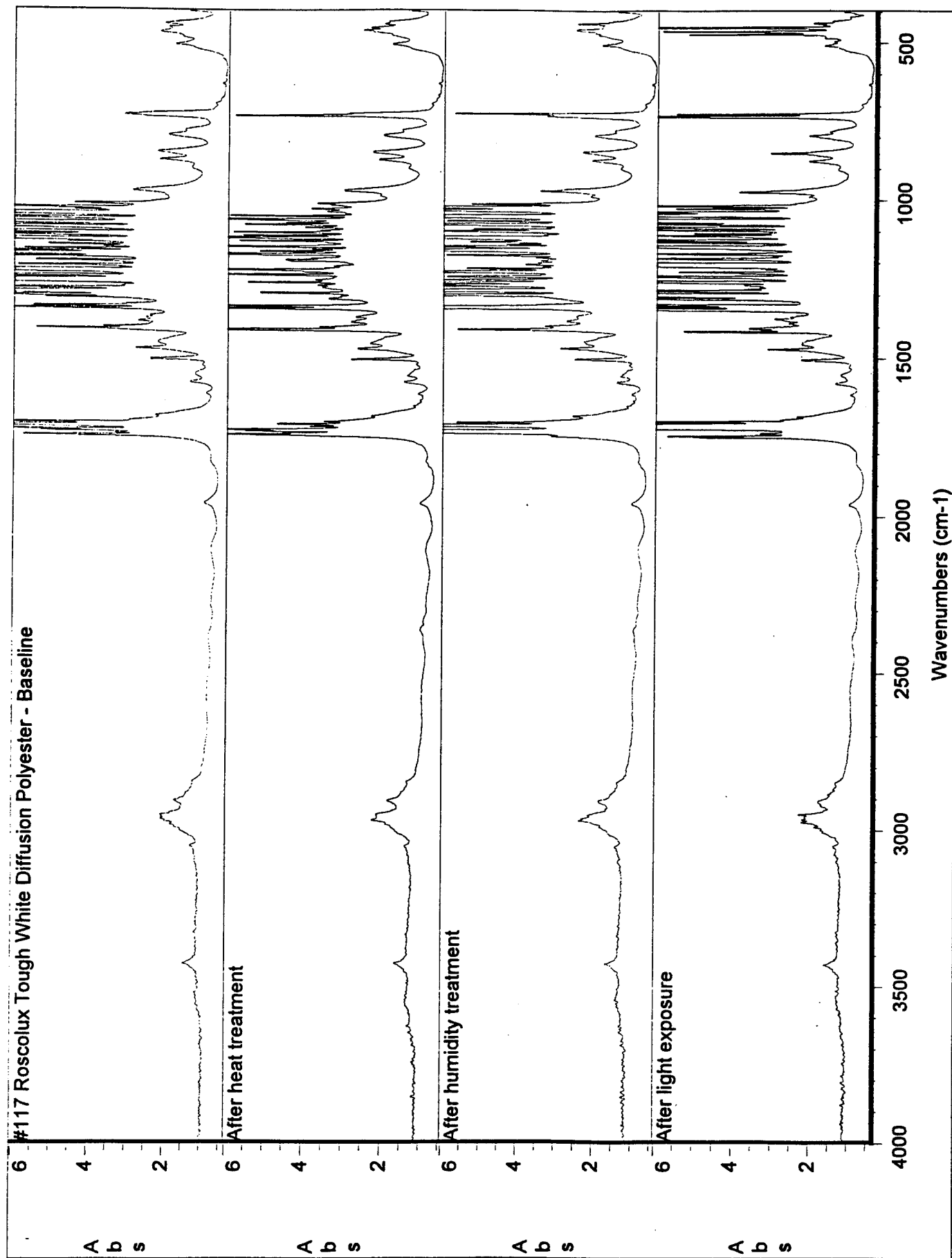


Figure 31. Infrared Spectrum of Filter 117 After Incubations.

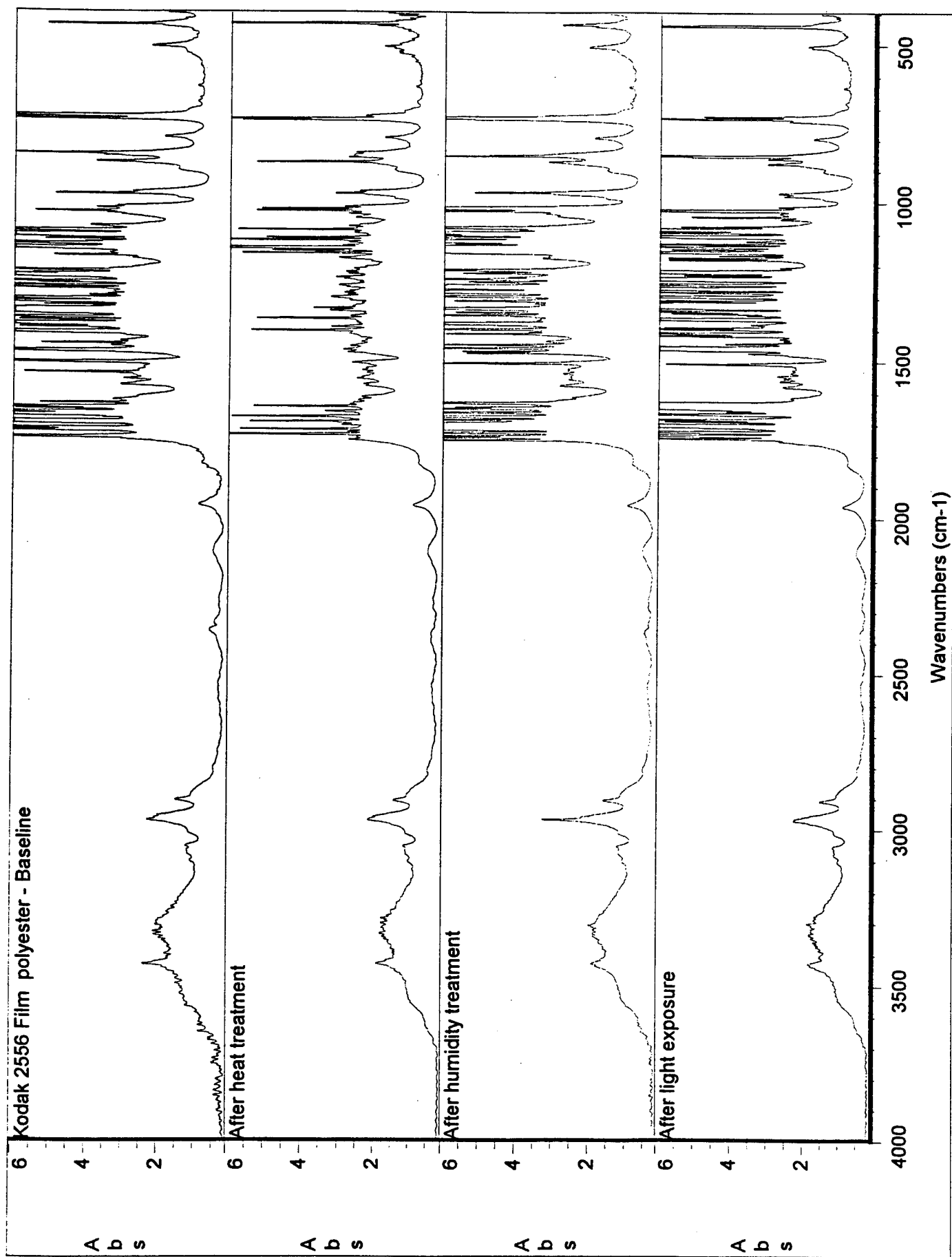


Figure 32. Infrared Spectrum of Filter 2556 After Incubations.

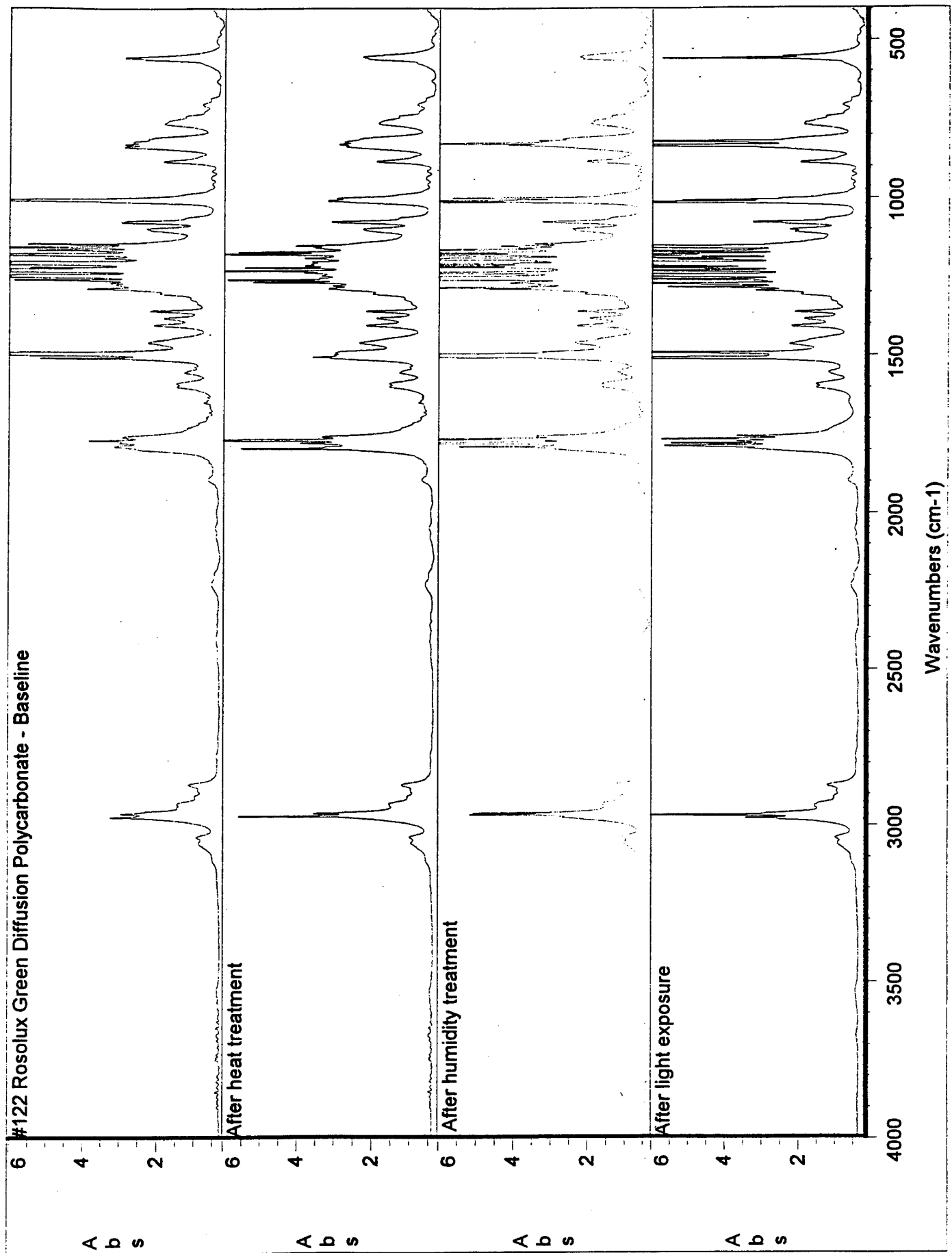


Figure 33. Infrared Spectrum of Filter 122 After Incubations.

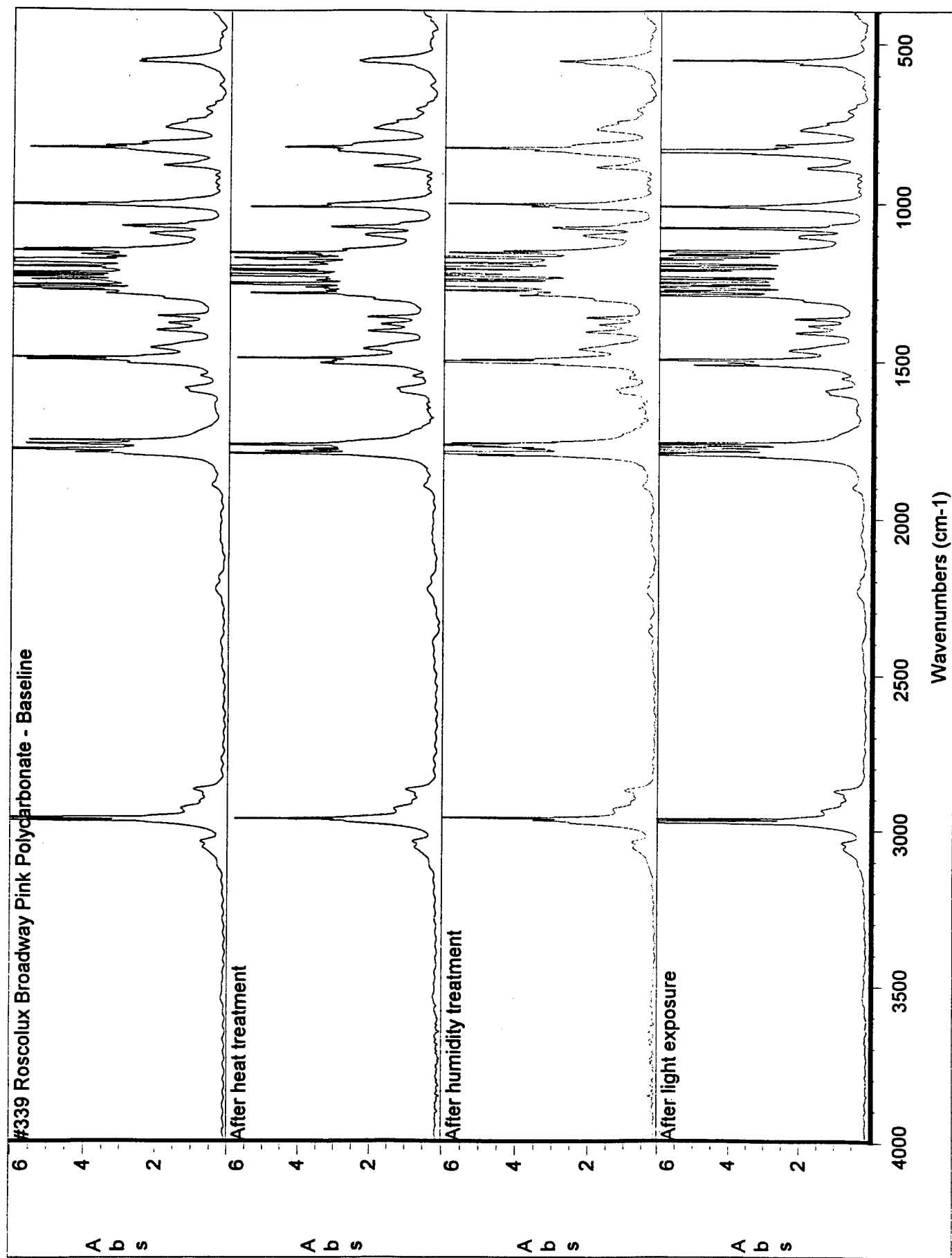


Figure 34. Infrared Spectrum of Filter 339 After Incubations.

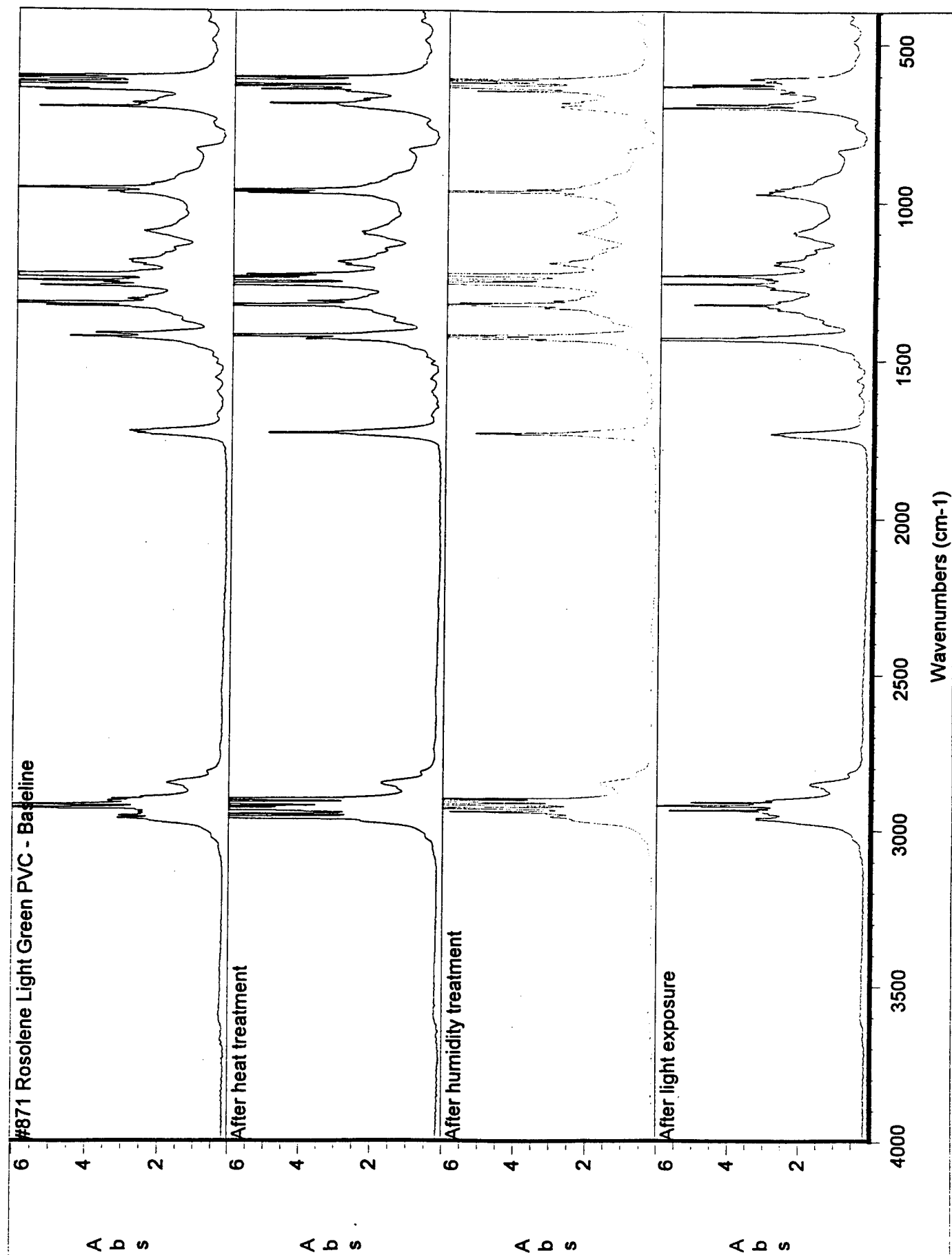


Figure 35. Infrared Spectrum of Filter 871 After Incubations.

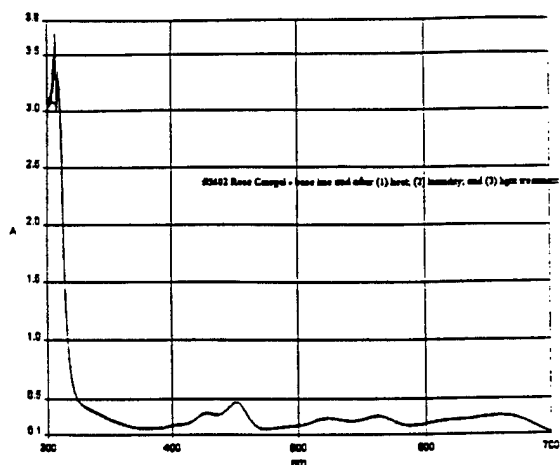


Figure 36. UV/VIS Spectra of Filter 3402 Before and After Incubations

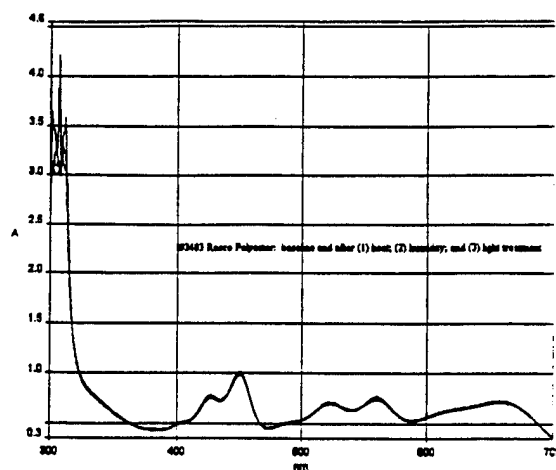


Figure 37. UV/VIS Spectra of Filter 3403 Before and After Incubations

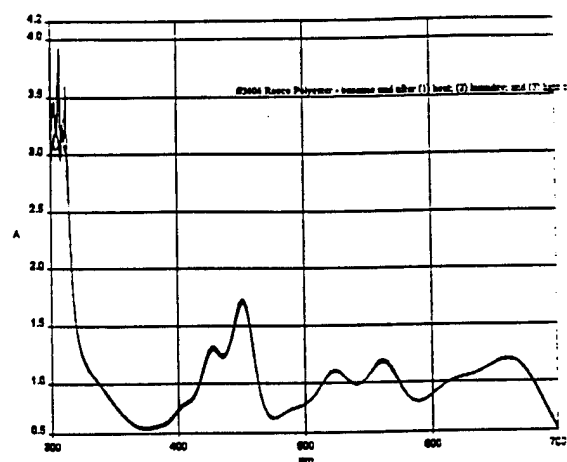


Figure 38. UV/VIS Spectra of Filter 3404 Before and After Incubations

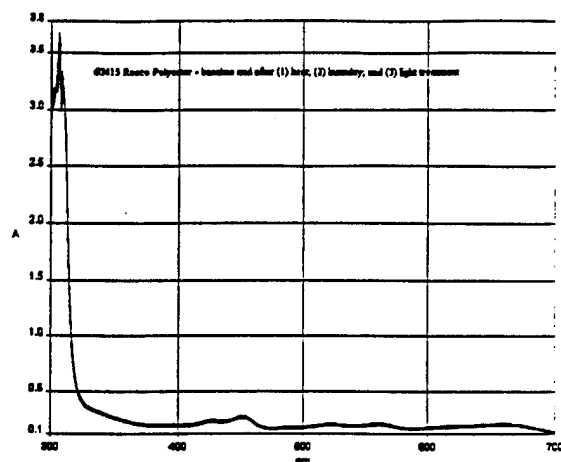


Figure 39. UV/VIS Spectra of Filter 3415 Before and After Incubations

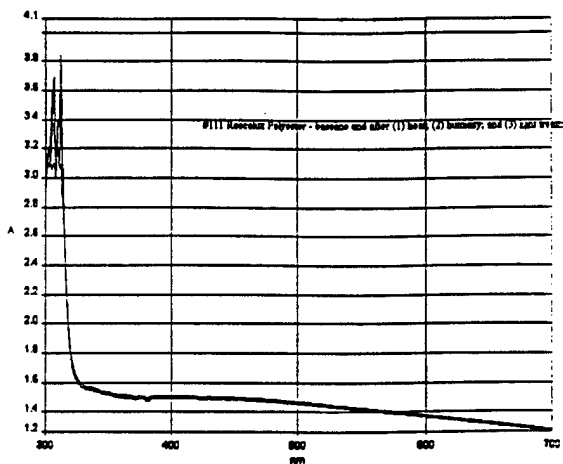


Figure 40. UV/VIS Spectra of Filter 111 Before and After Incubations

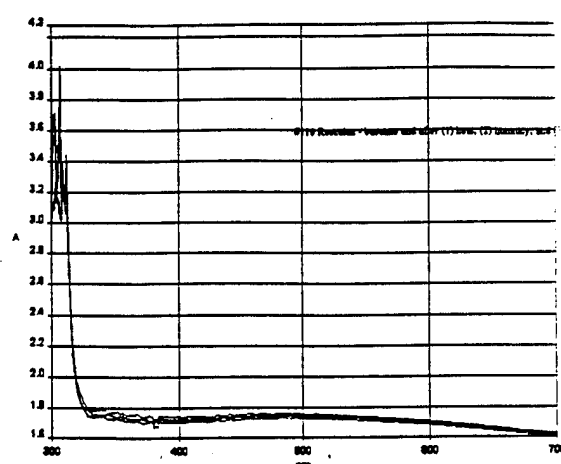


Figure 41. UV/VIS Spectra of Filter 116 Before and After Incubations

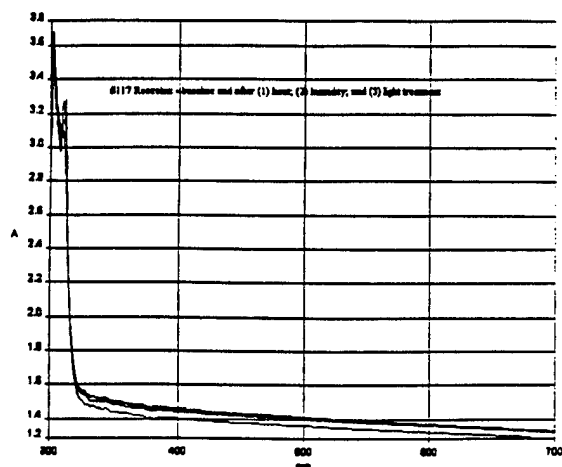


Figure 42. UV/VIS Spectra of Filter 117 Before and After Incubations

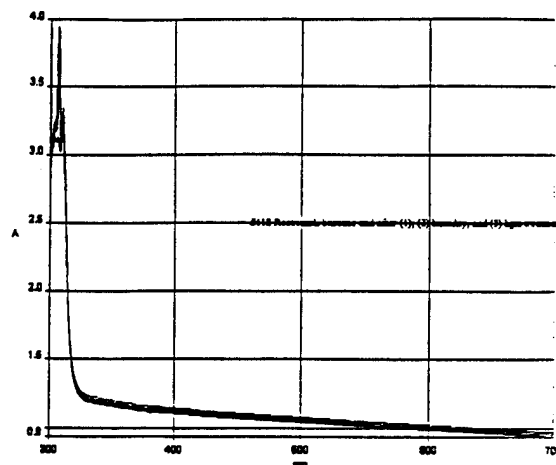


Figure 43. UV/VIS Spectra of Filter 118 Before and After Incubations

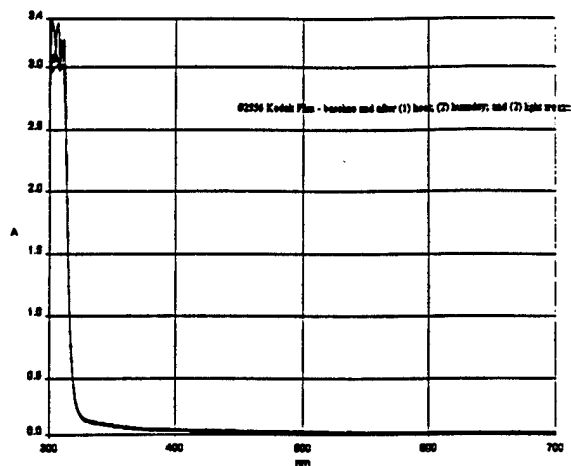


Figure 44. UV/VIS Spectra of Filter 2556 Before and After Incubations

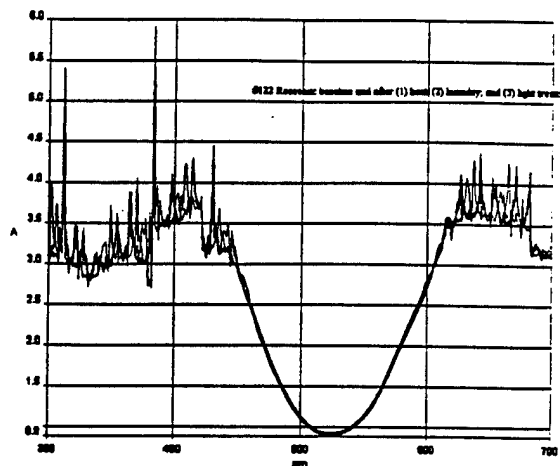


Figure 45. UV/VIS Spectra of Filter 122 Before and After Incubations

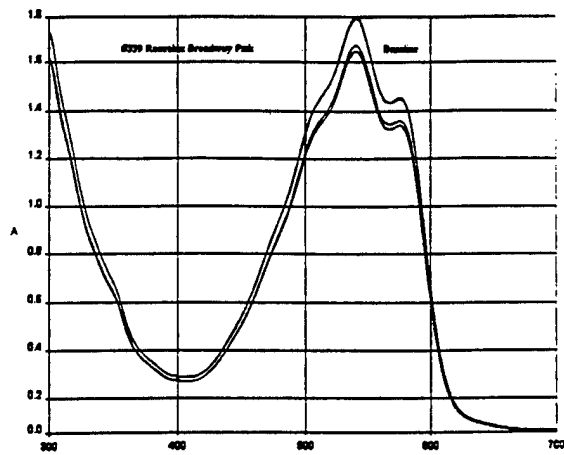


Figure 46. UV/VIS Spectra of Filter 339 Before and After Incubations

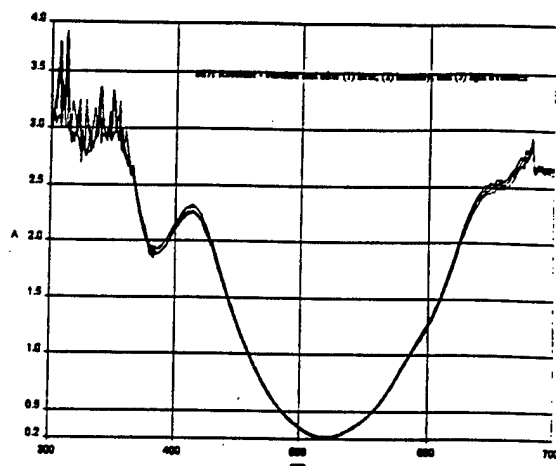


Figure 47. UV/VIS Spectra of Filter 871 Before and After Incubations

Table 1. Characteristics of Falant Filters

Filter Number	Description	Polymer matrix	Dye
3402	Rosco Cinegel	Polyester	Neutral density, N. 3
3403	Rosco Cinegel	Polyester	Neutral density, N. 6
3404	Rosco Cinegel	Polyester	Neutral density, N. 9
3415	Rosco Cinegel	Polyester	Neutral density, N. 15
111	Roscolux	Polyester	Tough Rolux
116	Roscolux	Polyester	Tough white diffusion
117	Roscolux	Polyester	Tough 1/2 white diffusion
118	Roscolux	Polyester	Tough 1/4 white diffusion
2556	Kodak	Polyester	0.04 Neutral density
122	Roscolux	Polycarbonate	Green diffusion
339	Roscolux	Polycarbonate	Broadway pink
871	Rosolene	Poly(vinyl chloride)	Light green

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